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THE EFFECT OF PRESSURES BELOW ONE ATMOSPHERE ON THE PERFORMANCE OF A PACKED COLUMN

A THESIS

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by

Leonard Rupert Daniel

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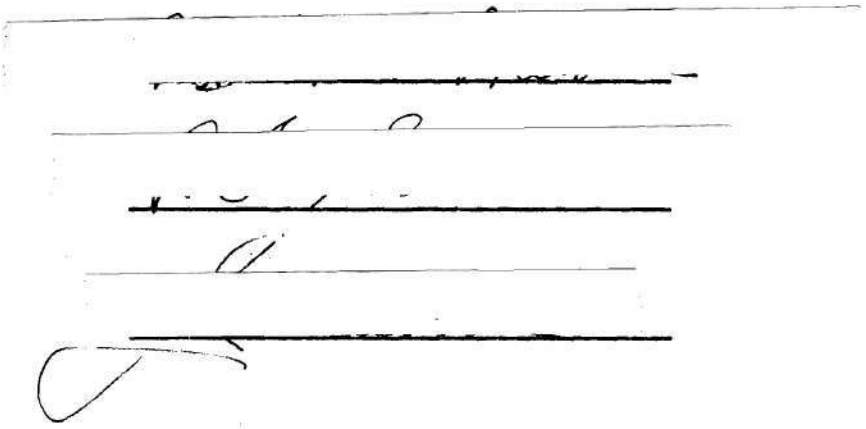
THE EFFECT OF PRESSURES BELOW ONE ATMOSPHERE

ON THE PERFORMANCE OF A PACKED COLUMN

Approved:



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Symbol	Term	Units
a	Surface area of packing	ft^2/ft^3
A	Cross sectional area	ft^2
d	Differential operator	Dimensionless
D	Distillate	$lb/secxft^2$
D_m	Diffusion Constant, molal units	mols/cm-sec
D_v	Diffusion Constant, volume units	cm^2/sec
F	Factor, equals $(G/L)^{1+P^{-0.35}}$	Dimensionless
G	Mass Gas Velocity	$lbs/sec-ft^2$
H	Column Height	ft
HETP	Height Equivalent to a Theoretical Plate	ft
HTU	Height of a Transfer Unit	ft
HTU_G	Height of Transfer Unit, gas film	ft
HTU_L	Height of Transfer Unit, liquid film	ft
HTU_{OG}	Overall Height of Transfer Unit, gas units	ft
HTU_{OL}	Overall Height of Transfer Unit, liquid units	ft
k_G	Gas film transfer coefficient	$lbs/sec-ft^2$
k_L	Liquid film transfer coefficient	$lbs/sec-ft^2$
K_G	Overall transfer coefficient, based on gas film	$lbs/sec-ft^2$
K_L	Overall transfer coefficient, based on liquid film	$lbs/sec-ft^2$

Symbol	Term	Units
L	Mass Reflux Rate	lbs/sec-ft^2
m	Slope of equilibrium curve	Dimensionless
M_m	Average Molecular Weight	gm or lbs
n	Number of even strips in Simpson's Rule	Dimensionless
NTU_{OG}	Number of Transfer units, based on overall gas units	Dimensionless
p, p_i	partial pressure, partial pressure at interface	mm of Hg. or atm.
P	Total pressure	mm of Hg. or atm.
q	Flow rate	gm/sec-cm^2
R	Universal Gas Constant	cm^3-atm/deg-mol
S	Cross-sectional Area	ft^2
t	time	sec
T	Temperature	^{o} K.
u, u_G, u_L	Velocity, gas and liquid velocities	cm/sec or ft/sec
w	Flow Rate	lb/sec
x, x_G, x_L	Film thickness, gas and liquid	cm or ft
x, x_i, x*	Liquid mole fraction, at interface, and corresponding to main body of gas	Dimensionless
x_D	Distillate mole fraction	Dimensionless
y, y_i, y*	Vapor mole fraction, at interface and corresponding to main body of liquid	Dimensionless
ρ, ρ_L	Gas, liquid density	gm/cm^3 or lb/ft^3

THE EFFECT OF PRESSURES BELOW ONE ATMOSPHERE ON THE PERFORMANCE OF A PACKED COLUMN

CHAPTER I

INTRODUCTION

The use of operating pressures below atmospheric in fractional distillation has assumed increasing importance in recent years. Some mixtures which under ordinary atmospheric distillation are separated with difficulty may in many cases be separated with ease when distilled at reduced pressures. Mixtures which decompose under atmospheric distillation may be separated safely when vacuum is employed, due to the resulting lower operating temperatures.

Many studies have been made in an effort to correlate the effect of various operating variables with the performance of packed fractionating columns--mostly at atmospheric pressure. Operating variables such as vapor velocity, packing material, over-all system composition, column diameter and height, and vapor-liquid ratio have been studied extensively.

The purpose of this investigation was to study column pressure (atmospheric and below) as an operating variable, and to correlate this pressure with column efficiency.

The general opinion of workers in the field of fractional distillation has been that a reduction in

operating pressure would result in a reduction in column efficiency. A paragraph taken from a paper presented before the Petroleum Division of the American Chemical Society in September 1944[1] reflects this belief:

> Even more important, perhaps, is the reduction of the interphase transfer of material in the spontaneous approach toward equilibrium that occurs at low pressures. The simple mass action law applies to the kinetics of this process. Hence, the rate of transfer is proportional to the densities of the two phases, other things being equal. The density of the vapor, in turn, is inversely proportional to the pressure, and this factor may be reduced a thousandfold or more in passing from atmospheric pressure to high vacuum conditions, reducing the efficiency of a column in the same way. Thus a fifty-plate column would realize only half a plate at 7.6 millimeters of Mercury if it could be used at all.

In a more recent publication[2], these same authors reported further study in the field of low-pressure distillation. In an attempt to improve separation efficiency at very low pressures, they designed a column which operated by "thermal" rectification; that is, rectification in which interphase transfer arises from the addition of heat to the reflux, or abstraction of heat from the vapor, or both, causing partial vaporization, partial condensation, or both, respectively. Their column consisted of two

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[1]Byron, E.S., Bowman, J.R., and Coull, J., Proceedings of the Petroleum Division of the American Chemical Society, September, 1944.

[2]Byron, E.S., Bowman, J.R., and Coull, J., Industrial and Engineering Chemistry, 43, 1002 (1951).

concentric tubes, the outer being heated, and the inner being cooled and rotated on its axis. Vapor rose in the annular space, and reflux flowed as a falling film on the inner surface of the outer tube. Partial vaporization of the reflux took place over the entire surface of the film, with partial condensation of the vapor stream occurring on the inner tube, and the condensate was thrown outward centrifugally as a spray to join the reflux stream.

These workers advocated the use of such a column for very low pressure work, due to the low efficiency to be expected in the usual "contact" rectification process, in which the interphase transfer arises from the spontaneous approach to equilibrium between the phases. They showed that theoretically the effect of pressure on column efficiency in the usual "contact" column is actually the sum of three effects--transverse diffusion, longitudinal diffusion, and the absolute vaporization rate--of which the transverse diffusion is favorable to decreased pressures, and the other effects are unfavorable. The overall result showed qualitatively that the efficiency decreased with the pressure.

However, workers in vacuum distillation have reported efficiencies in their work which compare favorably with work at atmospheric pressure.

Skoble and Driatskaya[3] reported increased efficiency

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[3]Skoble, A.I., and Driatskaya, Z.V., Neftyanoe Knoz., 24, No. 5, 39 (1946).

in the separation of certain hydrocarbons due to reduced operating pressures.

Nandi and Jalota[4], testing a 2.25 inch column packed to a height of 43 inches with copper lessig rings, reported efficiency at 250 millimeters of Mercury to be slightly below that obtained at atmospheric for low vapor velocities, but approaching the efficiency at atmospheric as vapor velocity was increased.

Smoker[5], in testing a rather unique multi-tubular packed column, found little difference in efficiency between runs at 760 and 200 millimeters of Mercury. He found a maximum efficiency for the atmospheric tests of 51 theoretical plates, while for the tests at 200 millimeters the maximum efficiency was 46 theoretical plates.

Schofield[6] tested a special rectangular packed tower 2.5 by 15 inches, at 760 and 180 millimeters of Mercury. The range of efficiencies was the same for both pressures, although, as should be expected, the ranges of throughputs for the two pressures were different. Expressed as "H.E.T.P." (Height Equivilant to a Theoretical Plate), his

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[4]Nandi, S.K. and Jalota, P.L., Transactions, Indian Institute of Chemical Engineers, 1, 57 (1947-48).

[5]Smoker, E.H., Transactions, American Institute of Chemical Engineers, 40, 105 (1944).

[6]Schofield, R.C., Chemical Engineering Progress, 46, 405 (1950).

range of efficiency was from 1.00 to 2.22 at 180 millimeters, and 0.96 to 2.54 at 760 millimeters.

Feldman, Myles, Wender, and Orchin[7] reported tests at pressures of 760, 150, 100, and 50 millimeters of Mercury. Although the range of tests at 760 millimeters was much wider than for the vacuum runs, the number of theoretical plates for these lower pressures was within the range covered by the atmospheric tests.

The first published paper which attempted a correlation of operating pressure with column efficiency--published while this investigation was in progress--was by Berg and Popovac[8]. These investigators used a 1 inch column, 2 feet high, packed with one-eighth inch stainless steel helices, and the test mixture, n-octane-toluene. For rates of through-put just short of flooding at each of eight pressures--20, 50, 100, 200, 300, 400, 640, and 760 millimeters of Mercury--these workers found that the efficiency of their column was essentially constant. A variation of 11 to 14 theoretical plates for the various pressures was obtained.

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[7]Feldman, J., Myles, M., Wender, I., and Orchin, M., Industrial and Engineering Chemistry, 41, 1032 (1949).

[8]Berg, L. and Popovac, D.O., Chemical Engineering Progress, 45, 683 (1949).

Struck and Kinney[9], in a paper published shortly after that of Berg and Popovac, reported data on four different packings, at pressures of 10, 20, 50, 100, and 740 millimeters of Mercury. Tests at 740 millimeters were made with the test mixture n-heptane-methylcyclohexane, whereas the vacuum tests were made with n-dodecane-cyclohexylcyclopentane. These investigators also attempted a correlation of operating pressure with column efficiency. Their results showed that at a constant boil-up rate at all the pressures studied, the efficiency of a given packing was essentially independent of operating pressure. On the basis of these results, Struck and Kinney claim agreement with Berg and Popovac. It should be observed, however, that the claim to agreement is not valid. The boil-up rate just short of flooding, as used by Berg and Popovac, was by no means constant, but decreased appreciably as operating pressures were lowered. On the other hand, data published by Struck and Kinney indicate that extrapolation of their results to boil-up rates just short of flooding for each of the pressures studied would result in a considerably improved efficiency at the lower pressures.

More recently, Myles, Feldman, Wender, and Orchin[10]

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[9]Struck, R.T. and Kinney, C.R., Industrial and Engineering Chemistry, 42, 77 (1950).

[10]Myles, M., Feldman, J., Wender, I., and Orchin, M., Industrial and Engineering Chemistry, 43, 1452 (1951).

made a study of column efficiency at reduced pressures, using berl saddles, triple and single-turn helices, glass spheres, and a commercial heli-grid packing. Their study was concerned with efficiencies at pressures of 20, 50, 100, 150, and 740 millimeters of Mercury. Their correlation was based on the average of the highest and lowest through-put possible at each pressure. Their results indicate a maximum efficiency for each packing studied in the neighborhood of 200 millimeters of Mercury with the efficiencies at 20 millimeters of Mercury essentially the same as those at 740 millimeters of Mercury. However, the authors admit that incomplete data prevents their results from being conclusive.

Thus, a survey of the published literature shows a considerable disagreement as to the effect of operating pressures on column efficiency.

CHAPTER II

DISTILLATION AND DIFFUSION THEORY

Literature Survey

The basic principle underlying the ability of a distillation column to separate partially a mixture of two miscible liquids is the fact that the vapor evolved from such a mixture is in general of different composition from that of the liquid. When such a mixture is partially volatilized, the vapor evolved is richer in the more volatile component, and the remaining liquid is correspondingly poorer. If this vapor in turn is partially condensed, the condensate is necessarily richer than the liquid originally volatilized, unless azeotropes or other anomalies are encountered. If vapor rising from the original mixture comes in contact with this condensate, an interaction takes place, resulting in partial condensation of this vapor, with evolution of a new vapor richer in the more volatile component. In a packed rectification column maintained at the boiling point of the liquid in the column, this partial vaporization and condensation takes place continuously up the column, with the packing providing the surface for the condensation and vaporization.

A study of the enrichment which takes place in a column therefore involves a study of mass transfer between the two phases, vapor and liquid. When material is trans-

ferred from one of these phases to the other across the interface separating the two, the resistance to the transfer in each phase causes a concentration gradient in each, with the two phases assumed to be in equilibrium at the interface between phases.

The "film" concept, first proposed by Whitman[1], is useful in attempting to analyze material transfer between phases. According to this theory, the region where most of the variation in concentration occurs in each phase is assumed to occupy only a small, comparatively negligible volume adjacent to the interface, this small volume being called a film. Further, the main resistance to transfer is assumed to be due to diffusion through the two films.

The fundamental equations for diffusion of gases were first derived by James Clerk Maxwell[2], and the derivation of equations of particular interest in rectification is listed below.

The basic equation for the unidirectional diffusion of a gas through a distance dx is given by

$$\frac{\partial \rho}{\partial t} = \frac{\partial}{\partial x}\left(D_v\left(\frac{\partial \rho}{\partial x}\right)\right)^* \qquad (1)$$

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[1]Whitman, W.G., Chemical and Metallurgical Engineering, 24, 147 (1923).

[2]Maxwell, J.C.: "Scientific Papers," Vol. 2, Cambridge University Press, Cambridge, England, 1890, p. 57.

*See page vii for meaning of symbols in this and following equations.

Since the rate of increase of the substance diffusing between the distances x and x + dx is equal to the rate of flow in and out of the two faces, it follows that

$$\frac{d\rho}{dt} = - \frac{dq}{dx}. \tag{2}$$

Therefore, integration of (1) under steady state conditions yields

$$q = - D_v \frac{d\rho}{dx}. \tag{3}$$

Consider two components, 1 and 2, diffusing in opposite directions. Since $q = \rho u$,

$$\rho_1 u_1 = - D_v \frac{d\rho_1}{dx} \quad \text{and} \quad \rho_2 u_2 = - D_v \frac{d\rho_2}{dx}. \tag{4}$$

Then, since $\rho = p/RT$, for component 1,

$$\frac{p_1 u_1}{RT} = - D_v \frac{d}{dx}\left(\frac{p_1}{RT}\right) = - \frac{D_v}{RT} \frac{dp_1}{dx}. \tag{5}$$

Under the condition that the total pressure of the system remains fixed, $p_1 u_1 + p_2 u_2 = 0$, from which, by elementary algebra,

$$\frac{u_1}{p_2} = - \frac{u_2}{p_1} = \left(\frac{u_1}{p_2}\right)\left(\frac{p_1 + p_2}{p_1 + p_2}\right) = \frac{u_1 - u_2}{P} \tag{6}$$

and

$$p_1 u_1 = - p_2 u_2 = \frac{p_1 p_2}{P}(u_1 - u_2). \tag{7}$$

Combining and rearranging equations (5) and (7),

$$\frac{dp_1}{dx} = - \frac{p_1 p_2}{D_v P}(u_1 - u_2), \tag{8}$$

and since $u = q/\rho = qRT/p$, it follows that

$$\frac{dp_1}{dx} = - \frac{p_1 p_2}{D_v P}\left(\frac{q_1 RT}{p_1} - \frac{q_2 RT}{p_2}\right) \tag{9}$$

$$= - \frac{RT}{D_v P}(p_2 q_1 - p_1 q_2). \tag{10}$$

In the simplified rectification theory, one of the basic assumptions is that of equi-molal overflow; that is, the number of moles of vapor rising from any given height in a column is equal to the number of moles of liquid reflux falling from the given height. In practice, when this assumption is not valid, equilibrium data is adjusted by using fictitious molecular weights so that equi-molal overflow does result or plate to plate calculations are made with a heat balance over each plate. Under this assumption,

$$q_1 = - q_2 \tag{11}$$

and equation (10) becomes

$$\frac{dp_1}{dx} = - \frac{RT}{D_v P} q_1(p_1 + p_2) = - \frac{q_1 P}{D_m}. \tag{12}$$

Integrating across the gas film, from 0 to x, and from

p_i to p, equation (12) yields

$$p_i - p = \frac{qP}{D_m}(x - 0). \tag{13}$$

Solving for q, and noting that $p/P = y$,

$$q = \frac{D_m}{x_G}\frac{(p_i - p)}{P} = k_G(y_i - y). \tag{14}$$

The derivation of similar equations for diffusion in liquids has been carried out[3], and the result may be expressed as

$$q = \frac{D_L}{x_L}(x_i - x) = k_L(x_i - x). \tag{15}$$

In studying the effect of operating pressure on the efficiency of a fractionating column, an examination of the two transfer coefficients, k_G and k_L, is useful.

As noted, the gas film transfer coefficient represents the ratio of the diffusivity constant, D_m, and the film thickness. Theoretical equations have been derived for determining the diffusivity constant[4], and these equations indicate that D_m is independent of the pressure, and approximately proportional to the square root of the absolute temperature.

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[3]Sherwood, T.K.: "Absorption and Extraction," McGraw-Hill Book Co., Inc., New York, 1937, p. 22.

[4]Sherwood, T.K., op. cit., p. 167.

Gilliland[5] studied experimentally the effect of pressure and other operating variables on the thickness of the gas film. By measuring the rate of vaporization of a number of liquids in a wetted-wall column through which air was forced at various velocities and pressures, he determined the effect of both pressure and velocity on the film thickness. Results showed that when variations in the Reynolds number were corrected for, the effect of pressure on the gas film thickness was negligible. The range of pressures studied was from 110 to 2,330 mm. of mercury. The effect of mass vapor velocity on the film thickness was appreciable, the film thickness being inversely proportional to approximately the 0.8 power of the mass velocity.

Thus it would appear that k_G is independent of the total pressure at a given mass velocity, but varies as the 0.8 power of the mass velocity, and approximately as the 0.5 power of the absolute temperature.

Due to the large variation of k_G with mass velocity, Chilton and Colburn[6] introduced the "Transfer Unit" concept by considering the ratio of k_G to the mass velocity as more nearly constant for varying velocities.

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[5]Gilliland, D.R., and Sherwood, T.K., Industrial and Engineering Chemistry, 26, 516 (1934).

[6]Chilton, T.H., and Colburn, A.P., Industrial and Engineering Chemistry, 27, 255-260 (1935).

A material balance over a section of column of height dH may be expressed as

$$dw = \frac{dyGS}{M_m}.^{*} \tag{16}$$

In terms of w, equation (14) may be expressed as

$$dw = k_G(y_i - y)dA = k_G\Delta y S dH a. \tag{17}$$

Combining and rearranging equations (16) and (17),

$$\frac{dy}{\Delta y} = \frac{k_G a M_m}{G} dH \tag{18}$$

which in integrated form may be written as

$$\int_{y_1}^{y_2} \frac{dy}{\Delta y} = \frac{k_G a M_m H}{G} \tag{19}$$

The integral on the left was defined by Chilton and Colburn[7] as the number of transfer units, which is analogous to the number of theoretical plates, used by Peters[8] in defining H.E.T.P., the height equivilant to a theoretical plate. The H.E.T.P. is found by dividing the height of the column

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*See page vii for Nomenclature.

[7]Chilton, T.H., and Colburn, A.P., op. cit., p. 255.

[8]Peters, W.A., Industrial and Engineering Chemistry, 14, 476 (1922).

by the number of theoretical plates. In the same way, H.T.U., the height of a transfer unit, is defined as the height of the packed section divided by the number of transfer units. The H.T.U. has a more sound theoretical basis for packed columns than the H.E.T.P. since it is based on a differential change up the column, whereas H.E.T.P. is based on finite steps.

The effect of the operating variables on the transfer coefficient for the liquid film, k_L, is difficult to analyze, due to the complicating effects of ionization, association, and dissociation, and the difficulty of eliminating natural convection.

Studies of the diffusivity constant for liquids, D_L, have not been very extensive. Early theoretical treatments on the evaluation of D_L by Sutherland[9], and Einstein[10], showed D_L to be directly proportional to the absolute temperature and inversely proportional to the liquid viscosity for a given liquid. A more recent publication[11] using the theory of absolute rates has shown the same relation between D_L, temperature, and viscosity.

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[9]Sutherland, W., Philosophical Magazine, 9, 781 (1905).

[10]Einstein, A., Annalen der Physik, 17, 549 (1905).

[11]Glasstone, S., Laidler, K.J., and Eyring, H.: "The Theory of Rate Processes," McGraw-Hill Book Co., Inc., New York, 1941, p. 516.

Many studies have been made on the relation between temperature and viscosity[12]; these studies show that an increase in temperature results in a decrease in viscosity for all liquids. Studies[13] also show that an increase in pressure results in an increased viscosity (with the exception of water), but this effect is not pronounced until relatively high pressures are attained. At pressures one atmosphere and below the effect of pressure on viscosity is negligible.

Published results showing the effect of operating variables on the thickness of the liquid film are lacking. However, absorption studies by Sherwood and Holloway[14], indicate that the absorption coefficient, k_L, increases with liquid rate.

Thus, it would appear that for a liquid mixture in a rectification column, the coefficient, k_L, is independent of pressure, but is a function of the liquid velocity and absolute temperature.

It is generally more convenient to work with overall coefficients in evaluating column performance, rather than

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[12]Partington, J.R.: "An Advanced Treatise on Physical Chemistry," Longmans, Green, and Co., London, 1951, pp. 95-110.

[13]Partington, J.R., op. cit., pp. 89-92.

[14]Sherwood, T.K., and Holloway, J.H., Transactions, American Institute of Chemical Engineers, 36, 30 (1940).

the individual coefficients, since interfacial conditions are usually not known. The overall coefficients are defined by the following equation:

$$dw = K_G(y^* - y) = K_L(x^* - x).^* \qquad (20)$$

The relation between individual and overall coefficients may be shown to be

$$\frac{1}{K_G} = \frac{1}{k_G} + \frac{1}{Hk_L}, \qquad (21)$$

where H is the Henry's Law constant. Sherwood[15] has shown for absorption of SO_2 by water that although increased temperature increases k_L because of the increased diffusivity, the Henry's constant, H, decreases with increased temperature. Sherwood states that "the net effect is not large in this case, although the liquid film resistance, $1/Hk_L$, is definitely increased by raising the temperature."

The relative resistance of the two films, liquid and gas, are of importance in estimating the performance of a rectification process. When Chilton and Colburn[16] first proposed the transfer unit concept for distillation, their derivation was on the assumption that the liquid-film

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*See page vii for meaning of symbols used.

[15]Sherwood, T.K., op. cit., p. 167.

[16]Chilton, T.H., and Colburn, A.P., op. cit., p. 255.

resistance was negligible. Johnstone and Pigford[17], studied the relative effect of the two resistances in a wetted-wall distillation column, with the systems, benzene-toluene, tolune-ethylene dichloride, ethanol-water, and acetone-chloroform. Their conclusions were that the liquid film resistance was negligible. Surowiec and Furnas[18] obtained data which showed that the liquid film resistance is much less than that of the gas. Their work was in a wetted-wall distillation column with the system ethanol-water.

In the same way that HTU was originally defined for the case in which only the gas film resistance was of importance, an overall HTU was defined by Colburn[19] to overcome the objection to the great variation of the overall transfer coefficients with gas and liquid velocities. The overall HTU, in units of y and y^*, is defined as the column height divided by the number of overall transfer units, NTU_{OG}, where

$$NTU_{OG} = \int_{y_1}^{y_2} \frac{dy}{y^* - y}. \qquad (22)$$

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[17]Johnstone, H.F., and Pigford, R.L., Transactions, American Institute of Chemical Engineers, 38, 25 (1942).

[18]Surowiec, A.J., and Furnas, C.C., Transactions, American Institute of Chemical Engineers, 38, 53 (1942).

[19]Colburn, A.P., Transactions, American Institute of Engineers, 35, 211 (1939).

*See page viii for table of Nomenclature.

Colburn also showed the relationship between the individual HTU's and the overall HTU to be

$$HTU_{OG} = HTU_G + (HTU_L)\ (mG/L) \qquad (23)$$

Equations (22) and (23) are derived for the case of equimolar counter diffusion which obtains in rectification columns.

Unfortunately, studies of HTU_{OG} in distillation columns have shown that this quantity also varies with gas and liquid velocities and perhaps other variables. Workers using relatively large packed columns have noted a decrease in HTU_{OG} with increased mass velocities. For example, Herman and Kaiser[20], in testing a 1 foot diameter column packed with fiber glass, reported a decrease in HTU with increasing vapor velocity, as did also Schofield[21]. Work with relatively small laboratory columns has, on the other hand, shown a decreased efficiency as vapor velocity is increased. Podbielniak[22] tested several columns varing in diameter from 5.7 millimeters to 13 millimeters in diameter, and noted a decrease in efficiency with increased reflux

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[20]Herman, A., and Kaiser, R.R., Transactions, American Institute of Chemical Engineers, 40, 487 (1944).

[21]Schofield, R.C., Chemical Engineering Progress, 46, 405 (1950).

[22]Podbielniak, W.J., Industrial and Engineering Chemistry, Analytical Edition, 13, 639 (1941).

rate. Struck and Kinney[23] experienced the same effect. Myles, et. al.,[24] experienced both an increase and decrease in HTU_{OG} in testing five different types of packing, the difference in efficiency depending upon the type of packing employed.

Colburn and Pigford[25] have attempted to predict the effect of various operating variables on HTU_{OG} for distillation columns by drawing upon information from the field of absorption, since, as they say, "the operations are analogous," and "distillation data are not reliable." These workers indicate that temperature has little if any effect on values of HTU_G, but that increasing temperature results in a marked decrease in values of HTU_L, because of the increased ratio of diffusivity to viscosity. As pointed out previously, however, Sherwood[26] cites a case of absorption of SO_2 in water in which the liquid resistance increases with temperature. Colburn and Pigford[27] further report

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[23]Struck, R.T., and Kinney, C.R., Industrial and Engineering Chemistry, 42, 77 (1950).

[24]Myles, M., Feldman, J., Wender, I., and Orchin, M., Industrial and Engineering Chemistry, 43, 1442 (1951).

[25]Colburn, A.P., and Pigford, R.L.; In "Chemical Engineers' Handbook," W.G. Perry, Ed., 3rd ed., McGraw-Hill Book Co. Inc., New York, 1950, p. 618.

[26]Sherwood, T.K., op. cit., p. 167.

[27]Colburn, A.P., and Pigford, R.L., op. cit., p. 619.

that the effect of pressure on HTU_{OG} is negligible, other than in determining the temperature, and thus the viscosity, of the liquid.

In view of the foregoing discussion it is apparent that the important variables to consider in evaluating column performance at reduced operating pressures are the gas and liquid velocities, provided that such quantities as column height and diameter, packing, composition, and reflux ratio are fixed.

It is interesting to note that in operating at reduced pressures the allowable liquid velocity decreases as pressure is reduced, whereas the allowable linear vapor velocity increases. An arbitrary rule of use in laboratory vacuum distillation concerning vapor velocity has been set up as follows[28]:

> The vapor velocity to be used under vacuum should be regulated so that it is related to the velocity used at atmospheric pressure by an inverse square root function of the fractional atmospheric pressure used. For example, if a pressure of 30.4 mm, or 1/25 atmosphere, is used the vapor velocity should not exceed the inverse of the square root of 1/25, or 5 times the velocity used under similar conditions at atmospheric pressure.

Another basis for selecting velocities at reduced pressures for comparison is a fractional value of the flooding velocities. Berg and Popovac[29] operated just

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[28]Carney, T.P., "Laboratory Fractional Distillation," The Macmillan Company, New York, 1949, p. 146.

[29]Berg, L., and Popovac, D.P., Chemical Engineering Progress, 45, 683 (1949).

below the flood point in a series of runs at several pressures, and used these velocities as a basis for correlation. Others[30] have chosen fifty per cent of the flooding velocity as basis. Myles, et. al.[31] chose the arithmetic mean of the highest boil-up rate possible short of flooding and the lowest measurable rate at the column reflux drip point. This basis was used rather than fifty per cent of flooding, because at some of the pressures studied, fifty per cent of the flood rate was so low as to be unmeasurable at the drip point.

New Theoretical Considerations

If, for a given column and packing operating under total reflux, the gas and liquid velocities are the variables of real significance, there might be reason to suspect that the time of travel of the gas and that of the liquid molecules through the column are important variables. The time for a gas molecule to travel the length of the packing is inversely proportional to the linear vapor velocity, and the time for a liquid molecule to travel the length of the packing is inversely proportional to the linear liquid velocity. The superficial vapor velocity, based on the entire column cross-section, although not the true velocity, is probably nearly proportional to it for

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[30]Myles, Feldman, Wender, & Orchin, op. cit., p. 1442.

[31]Ibid.

the range of velocities up to the flood point.

Assuming complete wetting of the packing at all reflux rates, so that the liquid covers essentially the complete column cross-section, a superficial linear liquid velocity may be defined as L/ρ_L. The liquid densities for liquids at their boiling point (orthobaric density) and pressures below one atmosphere are available for only a very few liquids. As an example of the effect of reduced pressure on this density, the density of ammonia liquid varies only about six per cent in going from 50 mm to 760 mm of mercury.[32]

Since orthobaric densities were not known for the liquids used in this investigation, and due to the slight variation in this density for liquids, it was assumed that u_L was approximately equal to L. Thus, although the mass reflux rate, L, is not a true measure of the linear liquid velocity, it is probably nearly proportional to it for the range of velocities usually investigated.

In considering the ability of a column to separate partially a mixture of two miscible liquids, one might associate with a molecule, or group of molecules, of higher boiling component rising as vapor from the still a probability of its passing from the vapor phase into the liquid

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[32]Lange, N.A.: "Handbook of Chemistry," N.A. Lange, Ed., 5th ed., Handbook Publishers, Inc., Sandusky, Ohio, 1944, p. 1462.

phase, and hence back to the still. In the same way, one might associate with a molecule, or group of molecules, of lighter boiling component descending as liquid from the top of the column a probability of its passing from the liquid phase to the gas phase, and hence back to the top of the column.

From elementary probability theory, it should be clear that the probability of both of these events occurring is the product of each of the individual probabilities.

Under the condition that the times of travel of liquid and gas through the packing are the only variables of consequence, the total probability would therefore be proportional to the product of the individual times. In terms of the velocities just discussed, the total probability would be inversely proportional to the product of the gas and liquid velocities, or under the assumptions made, proportional to the product $1/Lu_G$.

The higher this probability, the greater the separation in the column, and thus a greater efficiency, and a greater number of transfer units. Thus, HTU_{OG} should be a function of the product Lu_G, and should be independent of operating pressure or temperature.

If this were true, it would explain the arbitrary rule previously stated.[33] At a fixed operating pressure

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[33]Carney, T.P., op. cit., p. 146.

the vapor velocity is directly proportional to the liquid reflux rate. In addition, vapor velocity is inversely proportional to pressure for a fixed boil-up rate if temperature variation is moderate. Thus, at total reflux, permitting the vapor velocity to reach a value inversely proportional to the square root of operating pressure would result in a reflux rate directly proportional to the square root of pressure. Therefore, u_G would be inversely proportional to L, and the product Lu_G would be constant.

The effect of reflux ratio on HTU_{OG} has been studied by Colburn and Pigford[34], who state:

> There appears to be no reason why changing reflux ratio should affect the value of HTU_{OG} except possibly in changing the liquid flow rate and in changing the group mV/L and thereby the relative importance of gas and liquid velocities. Until separate values of HTU_G and HTU_L are known, the latter effect cannot be predicted.

However, as these authors point out, other workers, notably Furnas and Taylor[35], in a study with ethanol and water as test mixture, and Duncan, Koffolt, and Withrow[36], using five different test mixtures, found unexpectedly high values of HTU_{OG} for ratios of G/L higher than 1.5. More recently,

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[34]Colburn and Pigford, op. cit., p. 619.

[35]Furnas, C.C., and Taylor, M.L., Transactions, American Institute of Chemical Engineers, 36, 135 (1940).

[36]Duncan, D.W., Koffolt, J.H., and Withrow, J.R., Transactions, American Institute of Chemical Engineers, 38, 259 (1942).

Carter[37] in studying both a rectifying and stripping section of a distillation column, found HTU_{OG} to vary greatly as the ratio G/L increased. Colburn and Pigford[38] are of the opinion that at high ratios of G/L, "the liquor leaving the column is very close to equilibrium with the entering vapor, so that even a mild maldistribution would affect the countercurrent action of the different streams, and be reflected in high values of H.T.U."

Whatever the reason for the increased HTU_{OG}, the work of this investigation indicates that the effect is even more pronounced at reduced pressures than at atmospheric pressure.

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[37]Carter, W.L.: "Characteristics of a Packed Distillation Column: Correlations of HTU with Operating Variables for Rectification of Carbon Tetrachloride-Toluene Mixtures," Unpublished Thesis, Georgia Institute of Technology, 1950.

[38]Colburn and Pigford, op. cit., p. 619.

CHAPTER III

APPARATUS AND MATERIALS

The apparatus was made from non-corrodible material. All parts of the distillation column and auxiliary equipment were constructed of "Pyrex" glass, the brand name for a heat-resistant glass manufactured by the Corning Glass Company.

An assembly diagram of the apparatus is presented in Figure 11 in the Appendix.

1. Still and Column

The still consisted of a standard 5-liter, 3-neck distillation flask, with ground glass taper joints. The central neck was connected through an expansion joint to the column. One side neck was connected, through capillary tubing, condenser, and manometer, to the top of the main column condenser, in order to measure pressure drop across the column. The other side neck contained the bottom sample take-off line, and the product return line from the reflux divider at the top of the column.

The still was seated in a "Glas-col" heating mantel, purchased from Glas-Col Apparatus Company, Terre Haute, Indiana. Heat was supplied to the mantle by a 450-watt heating element inside the mantle. Another "Glas-col" heater covered the upper half of the still. The quantity of heat admitted to the mantles was regulated by variable

transformers, having a range of 0 to 135 volts.

The column was constructed of a 44-inch section of standard 45 millimeter diameter glass tubing, including standard ground glass taper joints attached at each end, of 45 millimeter diameter. A conical packing support was sealed to the column at a height 4 inches from the bottom taper joint. The column was constructed by the Ace Glass Company, Vineland, New Jersey.

The column was insulated with asbestos to a thickness of three-fourths of an inch. "Nichrome V" resistance wire, purchased from Leeds and Northrop Company, Philadelphia, Pennsylvania, was then wound about this first column insulation, with turns spaced one-fourth of an inch apart. Three such windings were made: one about the middle ten inches of packed section, and the other two above and below this middle winding to the ends of the column. The column was then covered with standard 3-inch Magnesia pipe insulation, approximately 1 inch thick.

Adiabatic operating conditions were maintained at each of the three column sections by supplying enough heat to the windings so that no heat would be transmitted radially through the column. Heat was supplied through variable transformers. The setting to be made on each of these transformers--one for each heater--was determined by a pair of thermocouple readings from two thermocouples; one located at the column wall, and a second located radially outward

from the first, and situated at the heater winding. When readings on these two thermocouples were the same--indicating the same temperature at the column wall and at the heater winding--no heat was being transmitted at that particular column height, and the transformer setting was correct. Such a pair of thermocouples was located at the middle of each of the three heater windings.

2. Column Packing

The packing used in this investigation was a special 8-turn glass helix, one-eighth inch in diameter and three-eighths inch long, manufactured by Ace Glass Company, Vineland, New Jersey. It seemed wise to test such a packing, since tests of a special 6-turn helix by Fenske[1] had shown a much lower efficiency than the usual single turn helix. The efficiency of the packing used in this investigation also showed a low efficiency compared to single turn helices, and other conventional types of packing, but compared favorably with the 6-turn helices previously mentioned. Table I in the Appendix shows a comparison of the data of this investigation with several standard packings.

Tests on the 8-turn helices showed a free volume of 83 per cent, and a static hold-up of 26 milliliters per foot of column. Approximately 10,000 individual pieces of

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[1]Fenske, M.R., Tongberg, C.P., and Quiggle, D., Industrial and Engineering Chemistry, 26, 1169 (1934).

packing were used to fill the column to the height desired, which was 30 inches.

The column was packed by dropping each piece of packing individually into the column, and the column was insured of proper packing by shaking the column frequently as the filling process was in progress.

3. Vacuum Pump

The pump employed to produce and maintain the desired operating pressures at the reduced pressures studied was a "Cenco Megovac Vacuum Pump," purchased from the Central Scientific Company of Chicago, Illinois. It operated on a voltage of 220 volts, and was guaranteed by the manufacturer to produce at the inlet a vacuum of 0.1 micron of mercury, or better.

4. Pressure Regulator

The operating pressure was maintained constant through use of a cartesian diver type of manostat.[2] The particular manostat used in this investigation was manufactured by Emil Greiner Company of New York. A sketch of this manostat is presented in Figure 1 on page 31. By reference to Figure 1, the principle and operation of the manostat

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[2]Gilmont, Roger, Industrial and Engineering Chemistry, Analytical Edition, 18, 633 (1946).

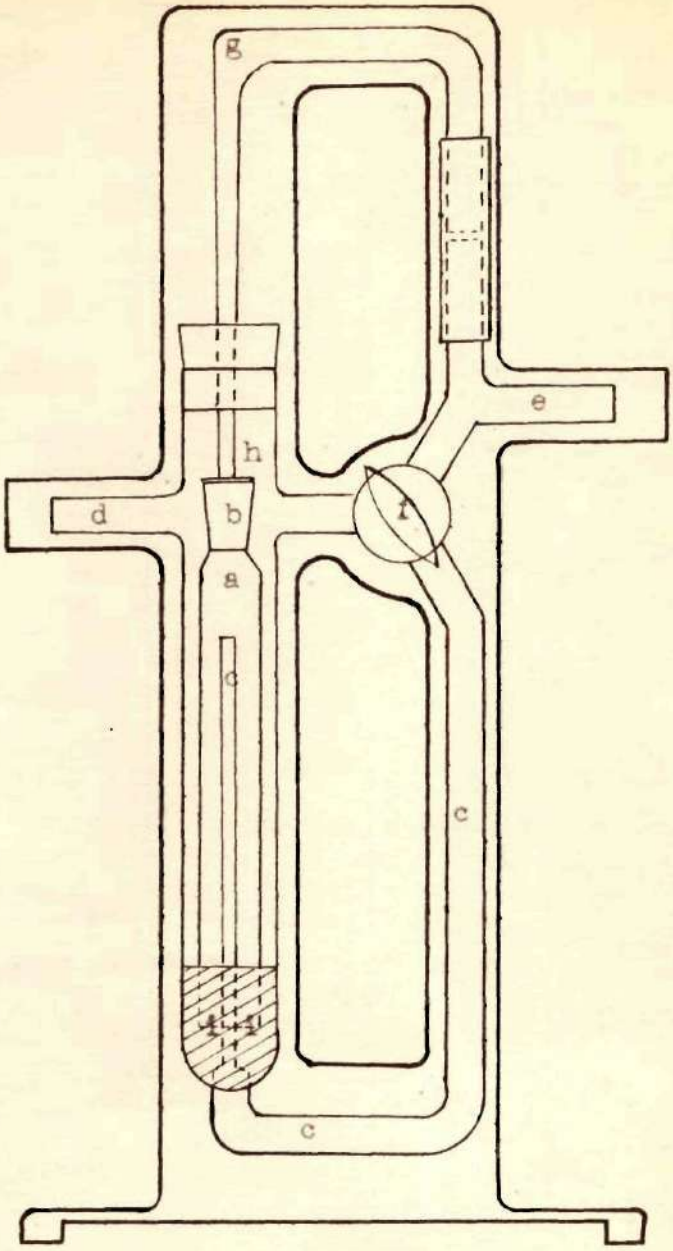

FIGURE 1

MANOSTAT

may be described. The system was evacuated to the desired pressure with stopcock "f" open so that the same pressure existed on both sides of the orifice "h" and inside the float "a" (the cartesian diver). The diver floated on the mercury "i". When the desired pressure was attained, stopcock "f" was closed. The pressure which was trapped in the float and line "c" controlled the pressure on the system side "d". So long as the pressure in the system remained the same as that trapped in the float, the rubber valve "b" sealed off orifice "h", and no gas was removed from the system. If, however, the pressure rose in the system through leakage, the float dropped, opening orifice "h" and allowing the vacuum pump to remove gas from the system through "d", until the system pressure again equalled that in the float. In actual operation the movement of the float was almost imperceptible, and the system pressure was maintained to within 0.1 mm. of mercury.

5. Reflux Divider

Product take-off was regulated by a reflux divider, shown in Figure 2 on page 33. The operation of the divider may be described by reference to Figure 2. Vapors from the column passed through the divider and into the main column condenser "a", where they were totally condensed and returned to the divider. The returning liquid passed over the lip "b", down the shaft "c", and over the ball and socket

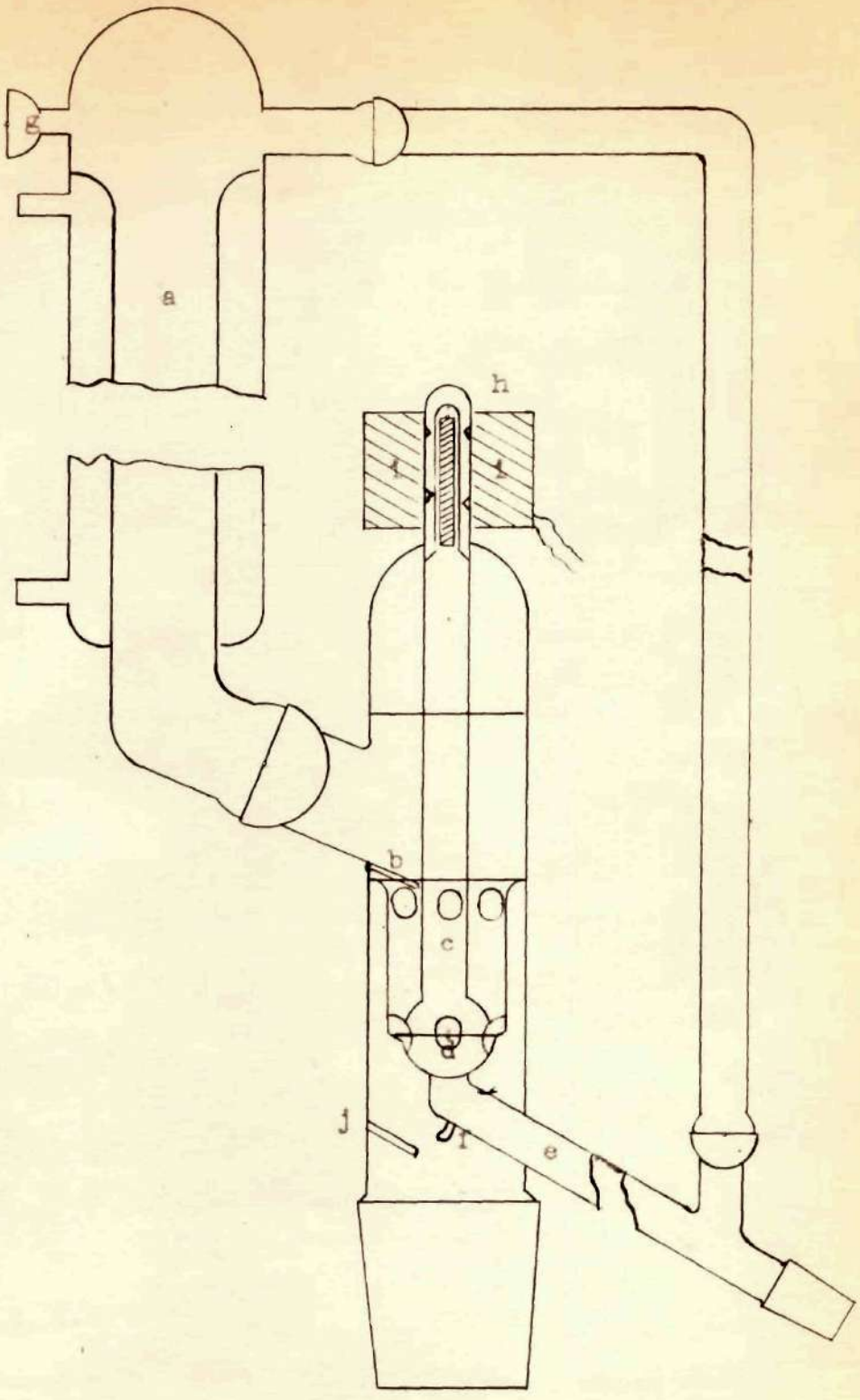

FIGURE 2 - REFLUX DIVIDER

joint "d". This joint opened and closed intermittently according to a setting on an electric timer. When the coil "i" was activated, the iron core "h" was raised, opening joint "d". When the joint was open, the condensate flowed out of the divider to the product take-off line "e". When the joint was closed, the condensate flowed over it, off the drip point "e", and back to the column. By regulating the time of activation of the timer, it was possible to maintain any desired reflux ratio. Vapor temperature was measured by a thermocouple in the well "j". Line "g" was connected to the pressure manometer to measure column pressure.

The reflux divider was constructed especially for this investigation by the Ace Glass Company of Vineland, New Jersey. The divider was insulated with asbestos, and was wound with a Nichrome V wire heater. This heater was connected to a variable transformer, so that the amount of heat admitted to the winding could be regulated so that no vapors were allowed to condense before reaching the main column condenser.

6. Electric Timer

An electric timer was employed to intermittently activate the ball and socket joint in the reflux divider. The timer used operated on a ten-second cycle, on a line voltage of 110 volts. It was manufactured by the General

Electric Company, and was listed by them as Timer Switch 3TSA144P10. The timer was so constructed that it was possible to regulate the activation time from 0 to 100 per cent of the cycle time by a simple setting on a scale with markings in per cent of cycle time.

7. Pressure Manometer

The pressure at the main column condenser was measured with absolute Mercury manometers. For pressures below 250 mm. of mercury, a manometer purchased from the Central Scientific Company of Chicago, Illinois, was used. This manometer was equipped with a movable mirrowed scale to eliminate parallax. For pressures above 250 mm. of mercury, an absolute manometer constructed by the author was used. Both of these manometers were tested for accuracy at low pressures with a McLeod gauge which was purchased from General Electric Company.

8. Pressure Drop Manometer

The pressure drop across the column (lengthwise) was measured by inserting directly into the line connecting the still with the main column condenser a U-tube manometer containing di-butyl phthalate. This liquid was chosen because of its low vapor pressure, and relatively low density. The difference in height between the two arms of this manometer was a direct measure of the column pressure drop.

The phthalate was changed periodically to avoid any error due to dilution.

9. Thermocouples and Potentiometer

Temperature measurements in this investigation were made indirectly by measuring the voltage generated in a thermocouple circuit, of which one thermocouple was located at the point at which temperature measurement was desired, and another thermocouple was immersed in an ice bath.

A pair of thermocouples was located at the middle of each of the three column heater windings--one at the column wall, and one at the heater winding. A thermocouple was located in the still, and in the reflux divider, and one was located at the column wall six inches below the top of the column to check on the temperature in this particular section of the column above the packing.

The thermocouples used consisted of an iron and a constantan wire twisted securely together in several turns. Thermocouple wire, of a duplex design, containing both the iron and constantan in a single strand, and insulated with glass fibers, was purchased from Leeds and Northrup Company of Philadelphia, Pennsylvania.

The voltage, in millivolts, induced in each thermocouple circuit was measured with a Double Range Precision Potentiometer Indicator purchased from Leeds and Northrup Company. The thermocouple circuits were connected to the

potentiometer through a Leeds and Northrup Ten-point Rotary Selector Switch, so that only one ice bath cold junction was necessary, and only one connection to the potentiometer for all nine thermocouple circuits was necessary.

10. Refractometer

Analysis of column equilibrium samples was made by index of refraction, and for this purpose a Bausch and Lomb modified Abbe type precision refractometer[3] was employed. In this type of refractometer, the prism hinge is vertical instead of horizontal, a monochromatic sodium D line is provided (no compensator system necessary), and the refractometer scale is horizontal and of the equidivision type with an accompanying calibration chart of scale reading versus refractive index. Because the scale has equal divisions, more accuracy per scale division is provided at the upper end of the scale where the change in refractive index per scale division is only one-third of the change at the lower end of the scale. The entire range of refractive indices is covered by two of these instruments. At 25° C. the two liquids used in this investigation had refractive indices on the lower end of the scale of the higher range instrument. Since the refractive index decreased with an

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[3]Weissberger, A.: "Technique of Organic Chemistry," Vol. I, Interscience Publishers Inc., New York, 1949, pp. 1209-1213.

increase in temperature, the temperature for analysis was raised to 43° C. to enable the upper end of the scale of the lower range instrument to be used. The increase in accuracy provided another significant figure in the analysis of samples.

The constant temperature of analysis, 43° C., was maintained by means of a constant temperature bath, manufactured by Precision Scientific Company of Chicago, Illinois.

11. Voltage Regulators

The amount of heat to the still mantles, the column, reflux divider, and bottom sample take-off heaters was regulated by means of variable transformers, called "Variacs," purchased from Superior Electric Company of Bristol, Connecticut. By use of these "Variacs," it was possible to vary output voltage continuously from no voltage up to a maximum of 135 volts.

Constant output from these "Variacs" depended upon a constant 115 volts being applied to the primary circuit. Since line voltage varied with power demand, it was necessary to supply the "Variacs" from a 3 KVA voltage stabilizer.

12. Binary Test Mixture

Methyl benzoate and isopropyl benzoate were chosen as the components of the test mixture used in investigating column efficiency at reduced pressures, because a mixture

was desired which could be used at both atmospheric pressure and reduced pressures as low as 10 millimeters of mercury. These chemicals appear to possess many of the preferred characteristics of a binary mixture for determining efficiencies in packed columns, which have been listed by Fenske[4]:

> The components should be obtainable in a high state of purity.
> The components should be relatively inexpensive if columns of large capacity are to be tested.
> The components should be of the same type as those regularly used in the columns.
> The mixture should be stable and non-corrosive in the columns.
> The mixture should allow condensation without resorting to refrigerative cooling.
> The mixture should be capable of easy and accurate analysis.
> The mixture should be as nearly ideal as possible, the components being completely miscible at all the temperatures used, no azeotropes being formed, and the relative volatility being substantially constant, for a given pressure, over the entire concentration range.
> The vapor-liquid equilibrium diagram should be available at the pressure used in testing, if a perfect solution is not formed and if the relative volatilities existing over the concentration range to be worked in are not known.
> The boiling points of the two liquids should be close enough so that, under the conditions of test, neither component is produced in a high state of purity. When this is the case, it is usually easier to obtain a more accurate analysis of the still and condenser samples. At the same time, the boiling points of the two liquids should not be so close that differences in pressure between the top and bottom of the fractionating section result in a considerable different number of plates calculated,

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[4]Fenske, M.R., Myers, H.S., and Quiggle, D., Industrial and Engineering Chemistry, 42, 649 (1950).

depending on whether the relative volatility existing at the top or the bottom of the section or some average value is used in making calculations. This factor is especially important when operating at reduced pressures, where the pressure drop through the fractionating section may be appreciable in comparison with the pressure at the top of the section.

Stumpe[5], who prior to the present investigation obtained vapor-liquid equilibrium data for the system methyl benzoate-isopropyl benzoate, showed that this test mixture follows most of the preferred characteristics listed. Isopropyl benzoate, however, appeared to decompose slightly upon continuous boiling at pressures above 200 mm. of mercury, since a yellowish color appeared in the still liquid. In this investigation, the still charge was changed periodically, and final runs with used still charge were checked with new still charge. Initial runs at 10, 50, 200, and 760 millimeters of mercury were made with fresh charges. Since a vapor sample rather than a liquid sample was taken from the still, the slight decomposition which took place in the still did not appear to affect the analysis of this sample, or of overall results.

The chemicals used were purchased at different times and bore the label of either the Eastman Kodak Company, or the Matheson Chemical Company. They were of the highest

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[5]Stumpe, N.E., Jr., "An Investigation of the Vapor-liquid Equilibrium at Pressures Below One Atmosphere for the System: Methyl Benzoate-Isopropyl Benzoate," Unpublished Thesis, Georgia Institute of Technology, 1950, pp. 4-5.

purity available. The purchased chemicals were further purified by distillation at 25 millimeters of mercury in the column of this investigation. The heart cut of a single distillation of methyl benzoate and a second heart cut from a double distillation of isopropyl benzoate were used in the column testing.

Physical data on the benzoates as reported in the literature are given in Tables III through XIII in the Appendix. Table IX in the Appendix shows vapor-liquid equilibrium data for each of the pressures used in this investigation, as reported by Stumpe.[6] Table X shows the comparison of this experimental equilibrium data with Raoult's Law.

Figure 10 in the Appendix shows the calibration curves of mole fraction of methyl benzoate versus refractometer scale reading at 43° C., which were used in the analysis of the test mixture.

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[6]Stumpe, N.E., Jr., op. cit., pp. 61-62.

CHAPTER IV

EXPERIMENTAL PROCEDURES AND RESULTS

Column Operation

The still charge for all experimental runs was 1500 milliliters of the test mixture, methyl benzoate-isopropyl benzoate, of composition 35 mole per cent methyl benzoate.

The initial step in making an experimental run was to bring the column and reflux divider up to the expected operating temperatures by admitting heat to the four heaters along the column and reflux divider. The quantity of heat was regulated by means of the variable transformers to which the heaters were connected. When the thermocouples along the column and reflux divider indicated temperatures near those to be expected under column operation, heat was admitted to the still heaters, and flow of cooling water was begun into the main column condenser.

At the same time, the vacuum pump was put into operation to evacuate the system to the desired operating pressure. The system was evacuated slowly to prevent any disturbance of packing, which could occur if gas were removed too rapidly. When the desired pressure was attained, the pressure regulator was set to maintain this pressure. This pressure was reached and set before the test mixture in the still began to boil.

Enough heat was admitted to the still heater to cause flooding conditions in the column. The column was allowed to remain at the flood point for about fifteen minutes, and then the heat was reduced. When flooding had ceased, heat was again increased until the column again began to flood. After fifteen more minutes, the heat to the still was adjusted for the particular boil-up rate desired.

Adiabatic column operation was attained by adjusting more closely the variable transformers on the column heaters, so that the heater temperatures, as measured by thermocouples, were essentially the same as the corresponding column temperatures. Approximately one hour was required--from the time the test mixture began to boil--to reach thermal equilibrium. After this time, only slight further adjustments were needed on the column heaters.

Heat to the reflux divider, and to the heater covering the upper half of the still, was regulated so that there was no condensation on the glass surfaces involved. Thus, no rectification took place except in the packed section of the column.

From three to six additional hours were required to reach fractionating equilibrium, depending upon boil-up rate and operating pressure. Equilibrium time could be predicted roughly as near the time when the temperature in the reflux divider remained constant. When this time was

reached, samples were withdrawn from the divider, at twenty minute intervals until analysis showed constant composition. When equilibrium was attained, thermocouple readings, heater voltages, and pressure drop were observed and recorded; a final sample was withdrawn from the divider, and a vapor sample from the still was withdrawn and condensed. These two samples were analyzed by means of the refractometer, and analyses recorded.

For total reflux runs, no distillate was removed, other than the few small samples for analysis.

The operation of the reflux divider has been described previously (see page 32). For runs other than total reflux, the column was started at total reflux, and operated as such for approximately one hour after thermal equilibrium was reached, after which the reflux divider was set for the desired reflux ratio. The column was then operated at this reflux ratio until equilibrium was attained.

Boil-up Rate-Pressure Drop Correlation

Measurement of the boil-up rate for each run was of prime importance, since both operating variables, vapor velocity and mass reflux rate, depended upon it. An easy method for quickly determining it was also desirable. Consequently, boil-up rate was measured indirectly during actual equilibrium runs by a previously determined correlation between the boil-up rate and pressure drop. Thus, during any given

equilibrium run it was easy to determine the boil-up rate by simply observing the pressure drop across the column and reading the boil-up rate from a graph.

In order to obtain this correlation, preliminary runs were conducted at each of the six pressures studied; for a given pressure drop across the column, the boil-up rate in grams per minute was measured by removing from the top of the column a timed distillate sample, and weighing it. Thus, by measuring this boil-up rate at several pressure drops, it was possible to construct a graph of boil-up rate versus pressure drop for each pressure. Figure 8 in the Appendix shows these calibration curves. These curves show that a logarithmic plot of pressure drop versus boil-up rate resulted in a straight line for each pressure, with lines for different pressures being essentially parallel. These curves are for total reflux operation. For finite reflux runs, the boil-up rate was set and measured before the reflux divider was put into operation.

A study of these curves shows that the pressure drop corresponding to a fixed boil-up rate increases greatly with succeedingly lower operating pressures. In the same way, the boil-up rate corresponding to a fixed pressure drop increases with increased operating pressure. In order to check on the relative compatibility of this data, a plot of operating pressure versus boil-up rate was constructed for fixed pressure drops of 5, 10, and 20 millimeters of di-

butyl phthalate. This plot is shown in Figure 9 in the Appendix. As indicated by these curves, the data for each pressure fell essentially on a single straight line for a fixed pressure drop. This type of plot was also employed by Struck and Kinney[1], with the same results.

After the completion of most of the equilibrium runs, a timed sample of distillate was removed, and its volume in milliliters measured, as a check on the calibration curves.

Analysis of Results

In this investigation an attempt was made to study the specific effect of operating pressure (atmospheric and below) on the performance of a packed rectification column, along with the operating variables vapor velocity, reflux rate, and reflux ratio. The results of this study are reported as values of the overall height of a transfer unit (gas units), HTU_{OG}, correlated against these operating variables.

Various runs were conducted at each of the following six pressures: 760, 400, 200, 100, 50, and 10 millimeters of mercury absolute pressure. In order that values of the transfer unit might be calculated for these pressures, it was necessary that vapor-liquid equilibrium data for the system studied be available. In conjunction with this

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[1]Struck, R.T., and Kinney, C.R., Industrial and Engineering Chemistry, 42, 77 (1950).

investigation, these data were obtained by Stumpe.[2]

For each of the aforementioned pressures, total reflux runs were conducted at various boil-up rates, ranging from approximately ninety per cent to fifteen per cent of the flood point.

Less extensive runs were conducted at finite reflux ratios for the pressures studied; reflux ratios (reflux to distillate) of 20:1, 10:1, 5:1, 2:1, and 1:1 were studied.

a. Reflux Rate - HTU_{OG} Correlation at Total Reflux

The experimental evidence of this investigation shows the height of transfer unit, HTU_{OG}, to be a function of the reflux rate at any given pressure. An increase in reflux rate resulted in an increased value of HTU_{OG}. The maximum allowable reflux rate was a function of operating pressure, the lowest allowable rate being experienced at the lowest pressure. Figure 3, page 48, shows the graph of reflux rate versus HTU_{OG} for each of the six operating pressures. These curves show that a logarithmic plot resulted in straight lines, with lines for different pressures being essentially parallel. It is interesting to note that for the range of reflux rates investigated--which at each pressure ranged from approximately ninety per cent to fifteen per cent of

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[2]Stumpe, N.D., Jr., "An Investigation of the Vapor-liquid Equilibrium at Pressures Below One Atmosphere for the System: Methyl Benzoate-Isopropyl Benzoate," Unpublished Thesis, Georgia Institute of Technology, 1950.

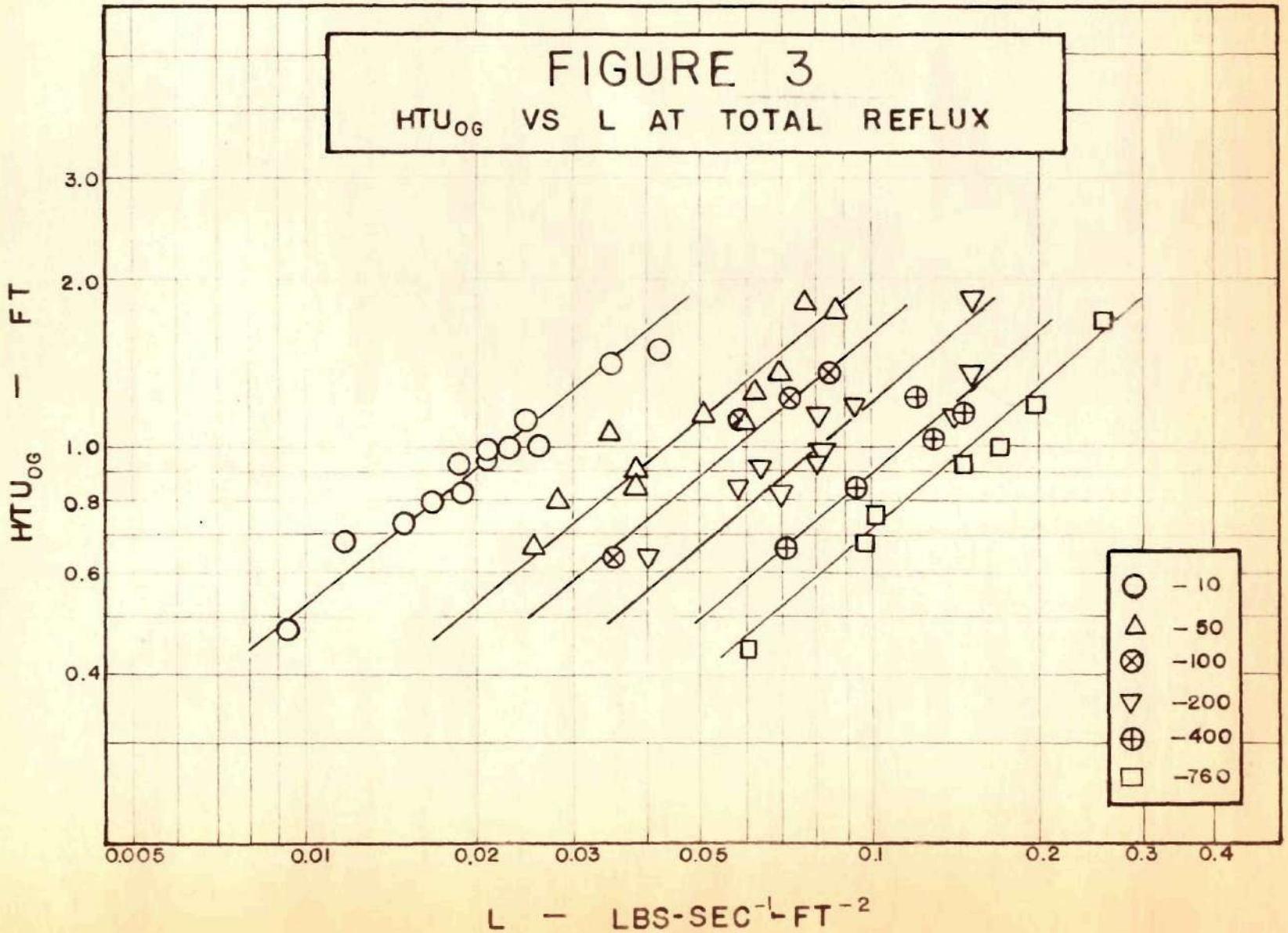
FIGURE 3
HTU$_{OG}$ VS L AT TOTAL REFLUX
HTU$_{OG}$ — FT
3.0
2.0
1.0
0.8
0.6
0.4
0.005
0.01
0.02
0.03
0.05
0.1
0.2
0.3
0.4
L — LBS-SEC^{-1}-FT^{-2}
-10
-50
-100
-200
-400
-760

flooding--the range of HTU_{OG} was the same for all pressures.

b. Vapor Velocity - HTU_{OG} Correlation at Total Reflux

When a column is operated at total reflux at a given operating pressure, the superficial vapor velocity, based on the complete column cross-section, is directly proportional to the reflux rate, or to the boil-up rate. For this reason, a plot of vapor velocity versus HTU_{OG} for a given operating pressure should result in the same type of curve as a plot of reflux rate versus HTU_{OG}. However, vapor velocity is almost inversely proportional to the pressure for a fixed reflux rate. Thus, it is expected that the order in which the curves for different pressures lie would not be the same with vapor velocity as abscissa as with reflux rate as abscissa.

Figure 4, page 50, which shows the plot of vapor velocity versus HTU_{OG} for each pressure, bears out the expectation that the order of curves would be different. In fact, the order of pressures is completely reversed in the two plots, Figure 3 and Figure 4.

c. HTU_{OG} - Lu_G Correlation at Total Reflux

From a study of the factors affecting column performance, and from a consideration of the probability of enrichment of a vapor stream passing up a rectification column, it was concluded in Chapter II that the product of the mass reflux rate and the linear vapor velocity is a good basis

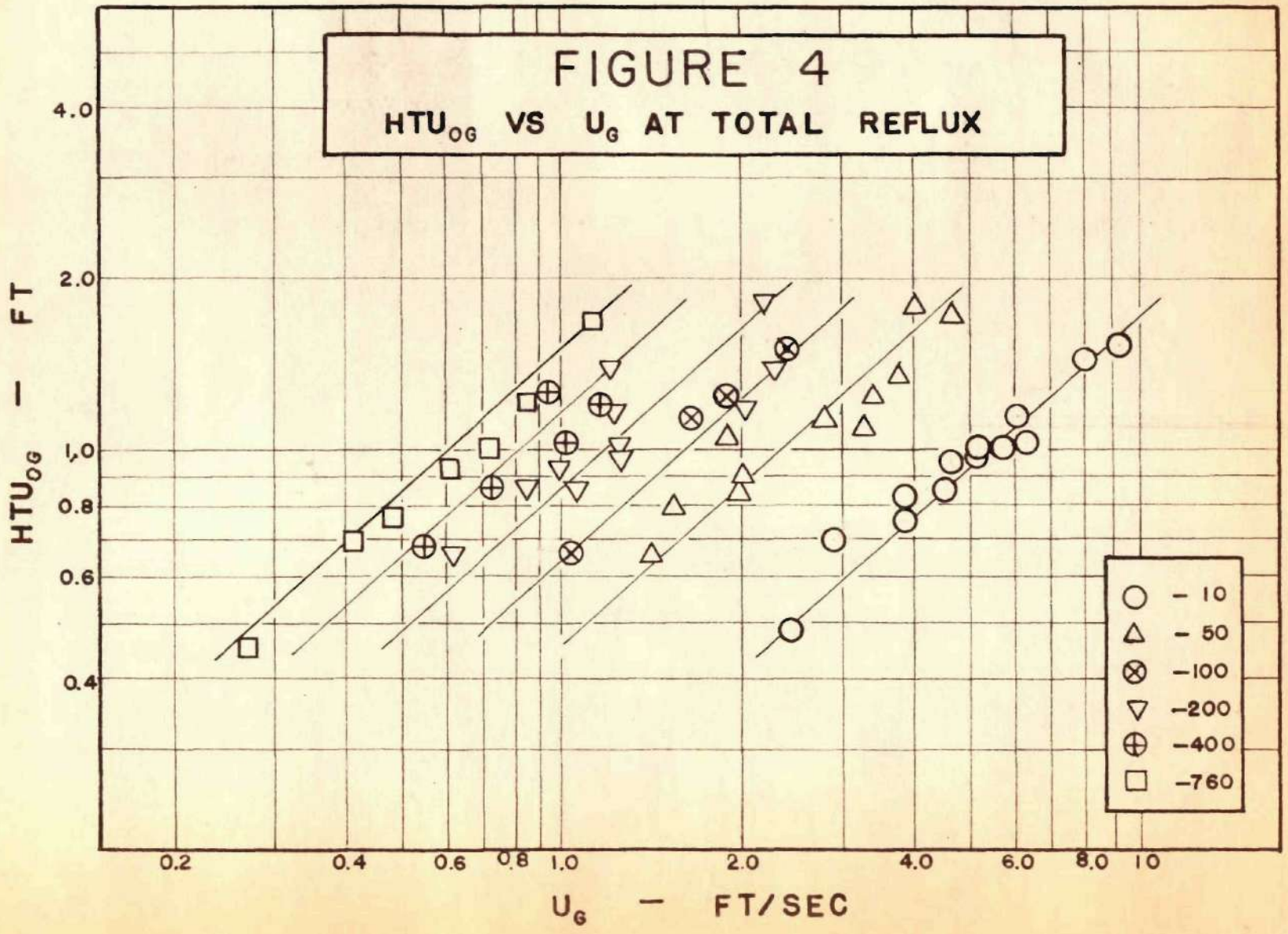
FIGURE 4
HTU$_{OG}$ VS U_G AT TOTAL REFLUX
HTU$_{OG}$ — FT
4.0
2.0
1.0
0.8
0.6
0.4
0.2
0.4
0.6
0.8
1.0
2.0
4.0
6.0
8.0
10
U_G — FT/SEC
- 10
- 50
-100
-200
-400
-760

for correlating efficiency data when operating at pressures below atmospheric. It was further concluded that with such a basis, column efficiency in terms of HTU_{OG} would be independent of the operating pressure and temperature.

In Figure 5 on page 52 is seen the plot of HTU_{OG} versus the product Lu_G for all the total reflux runs conducted at the six operating pressures studied. It is evident from this plot that, within the limit of the experimental error involved, HTU_{OG} is indeed independent of operating pressure for the correlation employed.

d. HTU_{OG} - $u_G\sqrt{P}$ Correlation at Total Reflux

An arbitrary rule, listed by Carney[3], for determining the optimum vapor velocity under reduced operating pressure, has been previously noted (see page 21). This rule states that the vapor velocity to be employed at reduced pressures to insure the same efficiency as at atmospheric distillation should be inversely proportional to the square root of the operating pressure. If the work of this investigation could be expected to follow this rule, a plot of HTU_{OG} versus the product of vapor velocity and the square root of pressure should result in only one curve for all the pressures studied. Figure 6, page 53, shows such a plot for the total reflux data of this investigation. This plot

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[3]Carney, T.P., "Laboratory Fractional Distillation," The Macmillan Company, New York, 1949, p. 146.

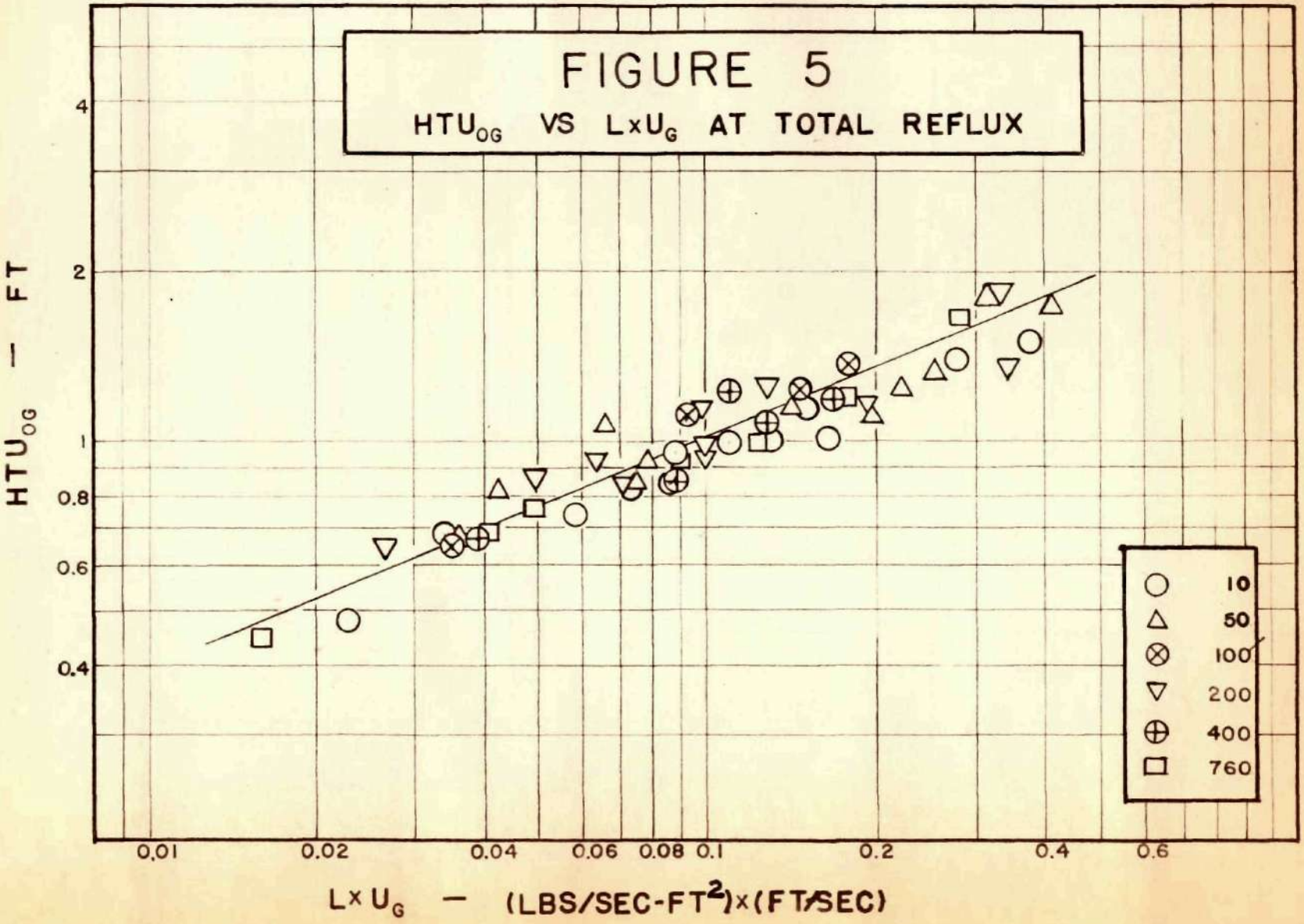


FIGURE 5
HTU$_{OG}$ VS L x U$_G$ AT TOTAL REFLUX
HTU$_{OG}$ — FT
4
2
1
0.8
0.6
0.4
0.01
0.02
0.04
0.06
0.08
0.1
0.2
0.4
0.6
L x U$_G$ — (LBS/SEC-FT^2) x (FT/SEC)
10
50
100
200
400
760

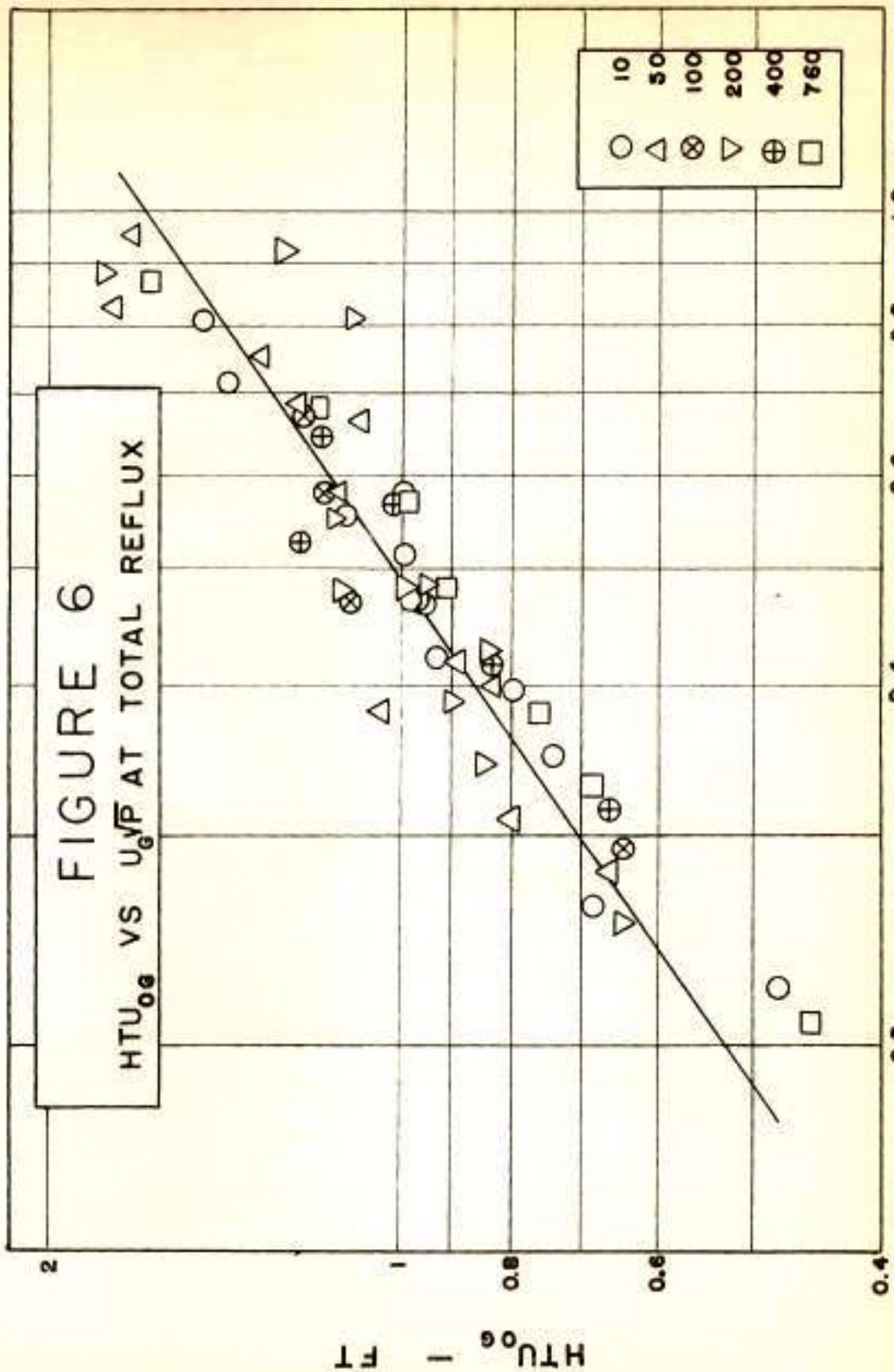


FIGURE 6
HTU$_{OG}$ VS $U_G\sqrt{P}$ AT TOTAL REFLUX
10
50
100
200
400
760
HTU$_{OG}$ — FT
2
1
0.8
0.6
0.4
0.2
0.4
0.6
0.8
1.0
$U_G\sqrt{P}$ — (FT/SEC)(ATM)$^{1/2}$

shows that the data does indeed fall essentially on one straight line for the logarithmic plot.

e. HTU_{OG} - F x Lu_G Correlation

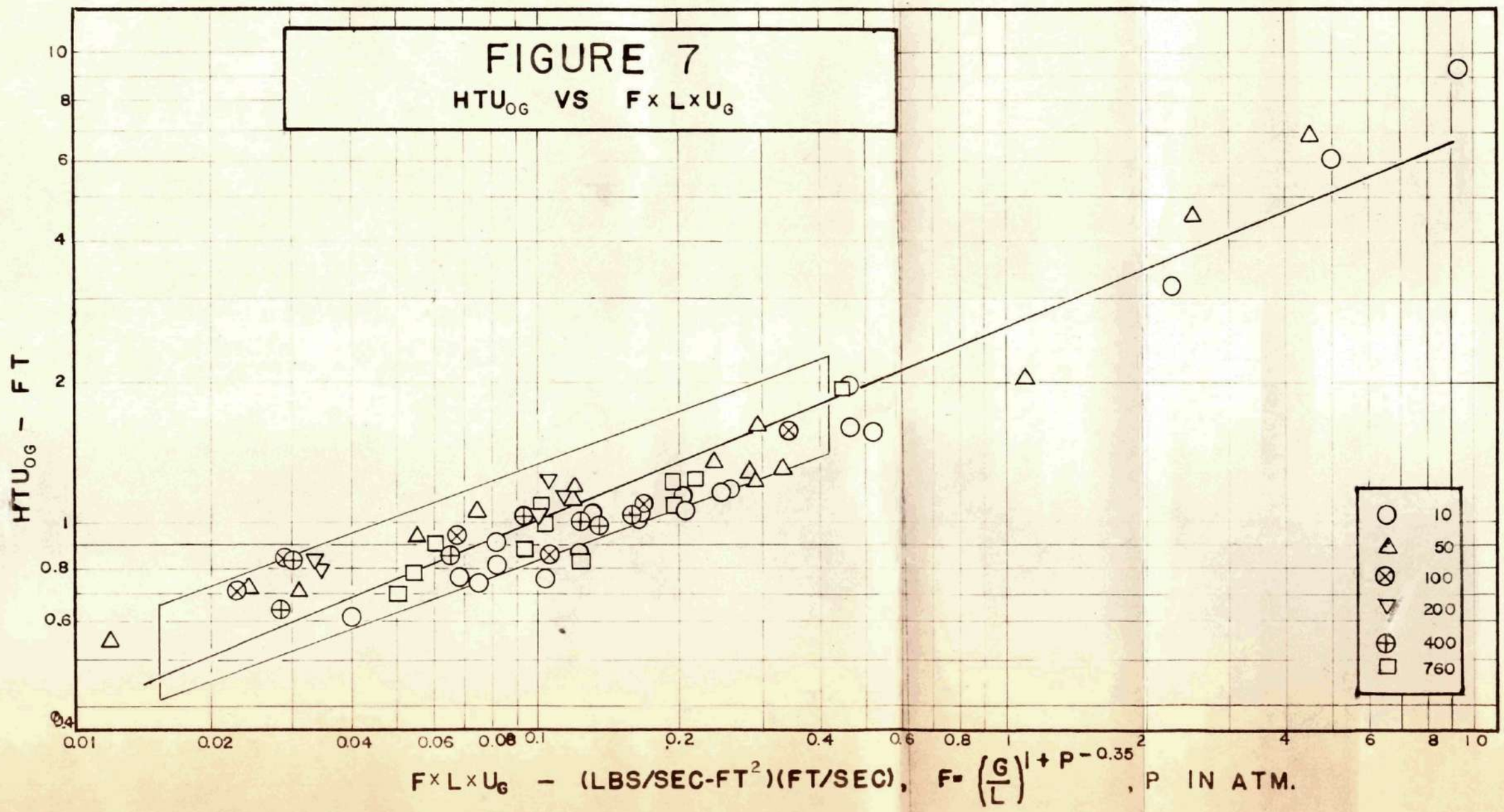
The data obtained for runs conducted at finite reflux ratios in this investigation indicated that not only was the efficiency a function of reflux rate and vapor velocity, but it was also a function of both the reflux ratio and the operating pressure. Consequently, a factor involving both the reflux ratio and pressure was sought to line up all data, both at total reflux and at the finite reflux ratios, on a single curve. It was found that multiplying the abscissa of Figure 5, Lu_G, by a special factor, F, where

$$F = \left(\frac{G}{L}\right)^{1 + P^{-0.35}}$$

(P measured in atmospheres) resulted in a single curve for all data obtained. Since, for total reflux, the ratio G/L is unity, the value of the factor, F, is also unity; therefore, a plot involving the factor, F, would be identical to Figure 5 for the total reflux data. Figure 7, page 55, shows the plot of HTU_{OG} versus the product of Lu_G and F for finite reflux ratios. In the interest of clarity, the total reflux data have been omitted from this plot, but all such data, if shown, would fall within the indicated parallelogram, and the straight line shown on Figure 7 is a continuation and duplication of the line shown in Figure 5.

FIGURE 7

HTU_{OG} VS $F \times L \times U_G$



$F \times L \times U_G$ – (LBS/SEC-FT^2)(FT/SEC), $F = \left(\frac{G}{L}\right)^{1} + P^{-0.35}$, P IN ATM.

Comparison with Published Data

a. Comparison of Column Packing

No published data are available in which the same size column and same test mixture as employed in this investigation were tested, but data are available for similar columns and test mixtures, and such data for several types of packing are listed in Table I in the Appendix. This comparison of atmospheric pressure data indicates that the 8-turn helices used in this investigation are less efficient than most common column packings, but are more efficient than the Fenske 6-turn wire helices.

b. Comparison with Data of Berg and Popovac

As noted in Chapter I, Berg and Popovac[1] were the first investigators to attempt to correlate distillation data at reduced pressures. These workers conducted tests at only one velocity for each pressure studied--the velocity just short of flooding.

In Table II in the Appendix their data is listed in terms of the product Lu_G, in which a relative value of the product ($Lu_G = 1$ at one atmosphere) has been calculated. As the data of Table II indicate, the value of this product for their data is substantially the same for each pressure

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[1]Berg, L., and Popovac, D.P., Chemical Engineering Progress, 45, 683 (1949).

with the variation apparently following the trend in variation of the number of plates. The variation in Lu_G is only slightly greater than the variation in number of theoretical plates. Since they concluded that the number of plates was constant, with the variation being due to experimental error, it should be reasonable to assume that the product Lu_G was also essentially constant.

CHAPTER V

CONCLUSIONS

From the results of this investigation the following conclusions can be made:

1. For the packing, column, and test mixture employed, HTU_{OG} increases with both liquid and vapor velocity for a given reflux ratio and operating pressure. For a fixed liquid or vapor velocity, HTU_{OG} is a function of operating pressure.

2. There is no record in the literature of a satisfactory or general correlation for distillation data for tests at both atmospheric pressure and reduced pressures. This investigation has shown that the product of mass reflux rate and linear vapor velocity is an excellent basis for correlating distillation data at pressures one atmosphere and below. With a plot of this product as abscissa and HTU_{OG} as ordinate, data at total reflux for all pressures and velocities studied fell on a single curve.

3. For a given finite reflux ratio, HTU_{OG} is a function of pressure, and at a given pressure, HTU_{OG} is a function of reflux ratio. Although tests at finite reflux ratios were not as extensive as at total reflux, these data were also correlated. When the product of a special factor,

F, where

$$F = \left(\frac{G}{L}\right)^{1 + P^{-0.35}},$$

the reflux rate, and the vapor velocity as abscissa and HTU_{OG} as ordinate, was plotted using logarithmic coordinates, a single straight line resulted for all the data of this investigation.

4. Eight-turn glass helices, one-eighth inch in diameter and three-eighths inch long, are less efficient than one-half inch berl saddles and one-fourth inch rings, but are more efficient than 6-turn wire helices.

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APPENDIX

SAMPLE CALCULATIONS

The following presentation is a sample of the data that were recorded for each run. The data listed are for Run 70, and the calculations of these data will be made in their entirety.

Run 70

February 12, 1951

Pressure (Millimeters of Mercury) .....10 mm.

Reflux Ratio (L/D) 5:1

Pressure Drop (Millimeters of di-butyl phthalate)..23 mm.

Temperatures

	Thermo-couple	Milli-volts	Tempera-ture, °C.
Still Liquid	1	5.083	96
Lower Column	2	4.590	87
Lower Heater	3	4.599	87
Middle Column	4	4.450	85
Middle Heater	5	4.450	85
Upper Column	6	4.220	80
Upper Heater	7	4.265	81
Top Column	8	4.231	80
Reflux Divider	9	4.169	79

Heaters	Volts
Still Bottom	50
Still Top	30
Lower Column	29
Middle Column	15
Upper Column	28
Reflux Divider	34

Analysis

	Refractometer Scale Reading	Mole % $C_8H_8O_2$ *
Still Vapor at Equilibrium	62.64	42.4
Overhead Vapor at Equilibrium	65.66	68.8

The boil-up rate in grams per minute was obtained by reading from Figure 8 in the Appendix the rate corresponding to the measured pressure drop. (The pressure drop for each run was observed under total reflux conditions, and then the reflux divider was set to regulate the reflux rate at the fixed boil-up rate.) As observed from Figure 8, the boil-up rate for Run 70 is 8.9 grams per minute.

Mass Boil-up Rate

The boil-up rate was converted into units of pounds per second per square foot of column cross-section through the following calculation:

$$G = \frac{(\text{gm/min})\ (\text{conversion factor})}{(\text{Cross-section Area})}$$

$$= \frac{(8.9\ \text{gm/min})\ (3.67 \times 10^{-5}\ \text{min-lbs/sec-gm})}{(0.0171\ \text{square feet})}$$

$$= 0.0191\ \text{lbs/sec-ft}^2$$

Mass Reflux Rate

The rate of flow of reflux was calculated by multi-

*See Figure 10 in the Appendix.

plying the boil-up rate by the ratio of reflux to boil-up, which for Run 70 was calculated to be:

$$L = (\text{Boil-up Rate})\ (L/G)$$

$$= 0.0191 \times 0.833$$

$$= 0.159 \text{ lbs/sec-ft}^2$$

Superficial Vapor Velocity

The superficial linear vapor velocity based on the total column cross-section was calculated on the basis of an average column temperature, which was taken to be the arithmetic mean of the inlet and outlet column temperatures. For Run 70:

Inlet Temperature - 80° C.

Outlet Temperature - 87° C.

Average Temperature - 83.5° C.

$$u_G = \frac{(G)\ (\text{ft}^3/\text{mol})\ (\text{Temperature Correction})}{(M_m)\ (\text{Pressure Correction})}$$

$$= \frac{(0.0191)\ (359)\ ([273 + 83.5]/273)}{(145)\ (10/760)}$$

$$= 4.7 \text{ ft/sec}$$

Number of Transfer Units

As defined by Chilton and Colburn[1], the number of

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[1]Chilton, T.H. and Colburn, A.P., Industrial and Engineering Chemistry, 27, 255 (1935).

transfer units corresponding to a given separation in a packed column is:

$$NTU_{OG} = \int_{y_1}^{y_2} \frac{dy}{y^* - y}$$

Since no analytical expression relating y^* to y was readily available, some method of approximate integration was desirable to evaluate the given difinite integral. For this investigation, that method known as Simpson's Rule[2] was employed. This method gives an exact value for definite integrals for which the integrand is a polynomial of degree three or less, and may be used to evaluate other definite integrals approximately to any desired accuracy.

The principle involved in Simpson's Rule is a consideration of the definite integral as a measure of area under a curve, in which the area is divided into n even strips of equal width, and the curve for each strip is approximated by a general cubic equation. By choosing n sufficiently large, any required accuracy may be obtained.

Preliminary investigation of the data obtained in this investigation indicated that use of n = 4 was sufficient for three significant figure accuracy, which was sufficient in terms of the experimental accuracy obtained.

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[2]Perry, J.L.: "Chemical Engineers' Handbook," J.L. Perry, Ed., 2nd ed., McGraw-Hill Book Co. Inc., New York, 1941, p. 235.

For n = 4, Simpson's Rule may be expressed as

$$\int_{y_1}^{y_2} f(y)\,dy = \frac{y_2 - y_1}{12}\left[f(y_1) + 4f(y_i) + 2f(y_{ii}) + 4f(y_{iii}) + f(y_2)\right]$$

where y_i, y_{ii}, and y_{iii} represent the y-values at the three middle divisions between strips.

Taking f(y) to be $(y^* - y)^{-1}$, the value of the definite integral will then be the number of transfer units.

The value of $y^* - y$ for various values of y was calculated by using the relation

$$y^* - y = (y^* - x) - (y - x).$$

Large-scale plots of $y^* - x$ were constructed for each pressure from the equilibrium data, which is shown in Table X in the Appendix. The value of $(y - x)$ was obtained from the relation between y and x expressed as the equation of the operating line

$$y = \frac{Rx}{R + 1} + \frac{x_D}{R + 1}$$

from which

$$x = \frac{(R + 1)y}{R} - \frac{x_D}{R}.$$

The steps involved in evaluating $y^* - y$ for each of the five y-values is shown in the following table (data from Run 70):

y	x	$y^* - x$	y - x	$y^* - y$
0.424	0.371	0.371	0.053	0.084
0.490	0.450	0.144	0.040	0.104
0.556	0.529	0.143	0.027	0.116
0.622	0.608	0.129	0.014	0.115
0.688	0.688	0.107	0.000	0.107

Having calculated the values of $(y^* - y)$, the following table shows the steps in evaluating the number of transfer units:

$(y^* - y)^{-1}$	Mult. Factor	Factor x f(y)
11.90	1	11.90
9.61	4	38.44
8.61	2	17.22
8.70	4	34.80
9.35	1	9.35
		$\sum$ = 111.71

$$(y_2 - y_1) = 0.688 - 0.424 = 0.264$$

$$NTU_{OG} = \frac{0.264 \times 112}{12} = 2.46.$$

Height of a Transfer Unit

The "Height of a Transfer Unit" is found by dividing the height of the column packing section by the number of transfer units. Accordingly,

$$HTU_{OG} = \frac{H}{NTU_{OG}}$$

$$= 2.50/2.46$$

$$= 1.02 \text{ feet.}$$

Product of Reflux Rate and Vapor Velocity

In correlating the distillation data for the various pressures studied, the product of the liquid reflux rate and the superficial linear vapor velocity was found to be useful. For the data of Run 70, this product is

$$Lu_G = (0.159)\ (4.7)$$

$$= 0.747.$$

Correlation Factor

It was found in this investigation that a plot of HTU_{OG} versus the product of Lu_G and a correlation factor, F, where

$$F = \left(\frac{G}{L}\right)^{1 + P^{-0.35}},$$

(P is here measured in atmospheres), resulted in data for all reflux ratios and operating pressures studied lying essentially on one curve. For Run 70,

$$\frac{G}{L} = \frac{R + 1}{R} = \frac{6}{5} = 1.2.$$

For 10 millimeters of mercury, pressure in atmospheres is 0.0132.

$$F = (1.2)^{1 + (.0132)^{-0.35}}$$

$$= 2.70$$

TABLE I

Comparison of Column Packing with other Packings

Packing	Packing Depth-ft	Column Diam-in.	Test Mixture	L, lb/hrxft^2	HTU_{OG} or HETP - ft
3/8" 8-turn glass helices[1]	2.5	1.77	Methyl Benzoate-Iso-pr. Benzoate	220	0.45
3/8" 8-turn glass helices[1]	2.5	1.77	Methyl Benzoate-Iso-pr. Benzoate	720	1.21
1/2" ceramic saddles[2]	8.5	2	n-Heptane-Methyl Cyclohexane	200	0.58
1/2" ceramic saddles[2]	8.5	2	n-Heptane-Methyl Cyclohexane	700	0.45
1/4" carbon rings[2]	8.5	2	n-Heptane-Methyl Cyclohexane	250	0.46
6-turn wire helices[2]	13.0	2.07	n-Heptane-Methyl Cyclohexane	700	1.53

[1]Author's Data

[2]Fenske, M.R., Tongberg, C.P., and Quiggle, D., Industrial and Engineering Chemistry, 26, 1169-1177 (1934)

TABLE II

Tabulation of Data of Berg and Popovac[3] in Terms of the Product L x u_G

Pressure mm Hg.	Boil-up Rate-cc/min	Avg. Col. Temp. °C.	Relative* L x u_G	Theo. Plates
20	1.80	-	1.15	13
50	9.3	44	1.28	13
100	12.7	59	1.27	13.5
200	18.7	76	1.45	12.5
300	20.8	87	1.23	11.5
400	25.8	96	1.45	11
640	26.9	111	1.03	11.5
760	28.6	118	1.00	11.75

*Ratio of L x u_G at given pressure to L x u_G at 760 mm.

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[3]Berg, L. and Popovac, D.O., Chemical Engineering Progress, 45, 683 (1949)

TABLE III

Vapor Pressures of Methyl Benzoate Below One Atmosphere[4]

Pressure mm. Mercury	Temperature °C
1	43.0
10	77.3
25	96.3
50	112.6
100	130.8
200	151.4
400	174.7
600	188.9
760	197.5

[4]Kahlbaum; Georg, W.A., Zeitschrift fur Physikalische Chemie, 26, 612 (1898)

TABLE IV

Vapor Pressures of Methyl Benzoate Below One Atmosphere[5]

Pressure mm. Mercury	Temperature °C
1	39.0
5	64.4
10	77.3
20	91.8
40	107.8
60	117.4
100	130.8
200	151.4
400	174.7
760	199.5

[5]Perry, J.L.: "Chemical Engineers' Handbook," J. L. Perry, Ed., 2nd ed., McGraw-Hill Book Co. Inc., New York, 1941, p. 161.

TABLE V

Specific Gravity of Methyl Benzoate and Isopropyl Benzoate[6]

	Temperature °C.	Specific Gravity (Basis: H_2O 25°C)
Methyl Benzoate	25	1.087
Isopropyl Benzoate	25	1.010

Density and Refractive Index of Methyl Benzoate[7]

Temperature °C.	Density g/ml	Refractive Index n_D
30	1.0796	1.5074
35	1.0721	1.5032
40	1.0690	1.4987

[6]Lange, N.A.: "Handbook of Chemistry," Handbook Publishers., Sandusky, 1944, pp. 532, 594

[7]Phadke, S.R., et. al., Journal Indian Chemical Society, 22, 235 (1945)

TABLE VI

Physical Constants for Methyl Benzoate and Isopropyl Benzoate[8]

	Methyl Benzoate	Isopropyl Benzoate
Mol. Weight, g/mole	136.14	164.20
Color of Liquid	colorless	colorless
Index of Refraction	1.51810	———
Density, g/ml	1.0937 1.088	1.0162
Melting Point °C.	-12.5	———
Boiling Point °C.	199.6	218.5

[8]Hodgman, C.D.: "Handbook of Chemistry and Physics," 30th ed., Chemical Rubber Publishing Company, Cleveland, 1947, p. 668

TABLE VII

Commercial Description of Isopropyl Benzoate[9]

"$C_6H_5COOCh(CH_3)_2$. Colorless liquid. M.W. 164.2; B.P., 218.5°C. @ 760 mm.; sp. g., 1.016 @ 15/4° C.; flash pt. (Cleveland Open Cup), 210° F.; Substantially insol. in water; sol. in alcohol and organic solvents; odor, pleasantly aromatic. Chemical properties; undergoes ester exchange using customary catalysts to form other esters; rapidly reactive source of the benzoyl radical; undergoes the usual reactions of esters on reduction, or other treatment. Uses: Alkyd resin manufacture to control cross-linking, chemical intermediate for manufacture of other esters such as benzyl benzoate; high boiling solvent for inks, dyes, resins; odorant. Available in commercial quantities. Carbide and Carbon Chemicals Div."

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[9]"New Chemicals for Industry," Chemical Industries, 67, 252 (1950)

TABLE VIII

Refractometer Calibration for Methyl Benzoate - Isopropyl Benzoate Mixtures at 43° Centigrade

Sample Number	Mole Percent Methyl Benzoate	Refractometer Scale Reading
1	0.00	59.17
2	12.86	60.05
3	24.40	60.97
4	35.64	62.00
5	46.33	63.05
6	56.10	64.06
7	65.98	65.27
8	75.22	66.55
9	83.84	67.85
10	92.24	69.37
11	100.00	70.81

TABLE IX

Smoothed Equilibrium Data[10]

Temperature °C.	Mole Percent Methyl Benzoate in Liquid	Mole Percent Methyl Benzoate in Vapor
At 10 mm. Mercury Absolute Pressure		
87.3	10.6	17.0
86.0	20.2	30.6
84.4	35.0	48.4
83.5	49.0	63.5
80.0	65.3	76.9
79.7	79.0	86.6
79.3	90.6	94.3
At 50 mm. Mercury Absolute Pressure		
128.2	10.6	16.1
125.6	20.2	29.3
124.0	35.0	46.8
122.5	49.0	61.5
120.9	65.3	75.3
115.8	79.0	85.5
119.1	90.6	93.8
At 100 mm. Mercury Absolute Pressure		
149.6	10.6	15.7
145.8	20.2	28.8
143.5	35.0	46.0
141.8	49.0	60.7
140.2	65.3	74.6
136.5	79.0	85.0
135.8	90.6	93.4

[10]Stumpe, N.E., Jr., op. cit., pp. 61-62

TABLE IX (Continued)

Temperature °C.	Mole Percent Methyl Benzoate in Liquid	Mole Percent Methyl Benzoate in Vapor
At 200 mm. Mercury Absolute Pressure		
171.1	10.6	15.3
165.6	20.2	28.4
165.0	35.0	45.3
163.0	49.0	59.8
162.0	65.3	73.9
159.1	79.0	84.5
157.0	90.6	93.2
At 400 mm. Mercury Absolute Pressure		
196.2	10.6	15.0
191.1	20.2	27.9
189.8	35.0	44.6
189.5	49.0	59.0
188.7	65.3	73.2
183.8	79.0	84.1
181.1	90.6	93.0
At 760 mm. Mercury Absolute Pressure		
223.1	10.6	14.6
220.5	20.2	27.5
217.3	35.0	44.0
216.7	49.0	58.2
213.8	65.3	72.6
210.0	79.0	83.7
208.5	90.6	92.8

TABLE X

Equilibrium Data -- Comparison with Raoult's Law[11]

Mole Percent Methyl Benzoate in Liquid	Mole Percent Methyl Benzoate in Vapor by Raoult's Law	Deviation of Raoult's Law Value from Smoothed Experimental Value
At 10 mm. Mercury Absolute Pressure		
10.6	18.6	1.6
20.2	32.8	2.2
35.0	52.1	3.7
49.0	66.8	3.3
65.3	79.0	2.1
79.0	88.0	1.4
90.6	96.0	1.7
At 50 mm. Mercury Absolute Pressure		
10.6	17.2	1.1
20.2	30.6	1.3
35.0	49.0	2.2
49.0	64.0	2.5
65.3	76.7	1.4
79.0	86.0	0.5
90.6	94.8	1.0
At 100 mm. Mercury Absolute Pressure		
10.6	16.5	0.8
20.2	30.0	1.2
35.0	48.0	2.0
49.0	63.0	2.3
65.3	76.0	1.4
79.0	86.0	1.0
90.6	95.0	1.6

[11] Stumpe, N.E., Jr., op. cit., pp. 63-64

TABLE X (Continued)

Mole Percent Methyl Benzoate in Liquid	Mole Percent Methyl Benzoate in Vapor by Raoult's Law	Deviation of Raoult's Law Value from Smoothed Experimental Value
At 200 mm. Mercury Absolute Pressure		
10.6	16.0	0.7
20.2	29.0	0.6
35.0	46.5	1.2
49.0	62.0	2.2
65.3	75.0	1.1
79.0	86.0	1.5
90.6	95.0	1.8
At 400 mm. Mercury Absolute Pressure		
10.6	15.5	0.5
20.2	28.2	0.3
35.0	45.0	0.4
49.0	60.8	1.8
65.3	74.5	1.3
98.0	85.0	0.9
90.0	93.8	0.8
At 760 mm. Mercury Absolute Pressure		
10.6	14.9	0.3
20.2	27.2	0.3
35.0	44.1	0.1
49.0	59.2	1.0
65.3	73.0	0.4
79.0	84.2	0.5
90.0	93.3	0.5

TABLE XI

Pressure Drop versus Boil-up Rate at Total Reflux

Pressure Drop mm Di-butyl Phthalate at 80 °F	Boil-up Rate grams/minute
At 10 mm. Mercury Absolute Pressure	
7	3.1
10	4.2
20	7.6
40	14.7
At 50 mm. Mercury Absolute Pressure	
4	5.75
10	13.0
15	18.4
22	25.1
32	33.5
At 100 mm. Mercury Absolute Pressure	
5	11.5
7.5	16.8
13	26.2
18	32.5

TABLE XI (Continued)

Pressure Drop mm Di-butyl Phthalate at 80 °F	Boil-up Rate grams/minute
At 200 mm. Mercury Absolute Pressure	
5	18.8
8	26.2
10	34.5
21	68.0
At 400 mm. Mercury Absolute Pressure	
7	40.0
10	54.5
12	65.0
20	96.0
At 760 mm. Mercury Absolute Pressure	
3.5	31.5
6	52.5
11.5	94.0
15	115.0
20	141.0

TABLE XII

Experimental Results -- Observed Data

Run	Pressure Drop -mm Di-butyl Phthalate	Equilibrium Data (Vapor) Inlet y_1	Outlet y_2	Column Temperature Inlet t_1 °C	Outlet t_2 °C
1	22.0	0.448	0.580	159	156
2	12.5	0.440	0.643	158	155
3	10.5	0.434	0.658	159	156
4	16.0	0.452	0.666	138	135
5	12.5	0.435	0.673	138	135
6	7.0	0.490	0.657	160	156
7	25.0	0.475	0.645	140	137
8	12.0	0.500	0.685	183	179
9	9.5	0.430	0.700	184	180
10	7.0	0.416	0.685	184	180
11	5.0	0.435	0.746	182	178
12	4.0	0.426	0.735	184	178
13	7.0	0.441	0.696	181	177
14	10.0	0.416	0.643	183	178
17	12.5	0.462	0.734	139	135
18	6.0	0.457	0.740	138	134
19	15.0	0.451	0.670	142	137
20	9.0	0.430	0.690	139	135
21	5.0	0.439	0.744	139	135
22	27.0	0.458	0.687	122	118
24	12.5	0.462	0.736	121	117

TABLE XII (Continued)

Run	Pressure Drop - mm Di-butyl Phthalate	Equilibrium Data (Vapor) Inlet y_1	Outlet y_2	Column Temperature Inlet t_1 °C	Outlet t_2 °C
26	24.0	0.451	0.700	122	119
28	12.5	0.437	0.702	121	116
29	41.0	0.440	0.750	91	81
30	20.5	0.455	0.765	86	81
31	12.5	0.405	0.821	85	79
32	21.0	0.412	0.775	87	79
33	33.0	0.455	0.716	90	82
34	27.0	0.455	0.733	86	80
35	18.0	0.498	0.744	88	81
36	30.0	0.420	0.680	90	81
42	18.0	0.450	0.695	123	117
43	26.0	0.467	0.765	88	82
44	33.0	0.416	0.610	123	118
45	27.0	0.425	0.605	124	120
46	4.0	0.415	0.815	119	115
47	12.0	0.417	0.676	121	118
48	15.0	0.402	0.633	121	118
49	6.0	0.413	0.740	120	116
50	21.0	0.413	0.620	124	118
51	12.5	0.413	0.635	123	117
52	6.5	0.415	0.721	122	116
53	10.0	0.385	0.655	140	134

TABLE XII (Continued)

Run	Pressure Drop - mm Di-butyl Phthalate	Equilibrium Data (Vapor) Inlet y_1	Outlet y_2	Column Temperature Inlet t_1 °C	Outlet t_2 °C
54	8.0	0.375	0.730	122	116
55	27.0	0.385	0.600	124	117
56	24.0	0.397	0.608	122	116
57	10.0	0.400	0.691	122	116
58	12.5	0.390	0.685	120	116
59	16.0	0.405	0.800	88	81
60	23.5	0.490	0.788	88	80
61	26.5	0.456	0.740	87	80
62	14.0	0.415	0.770	88	80
63	26.5	0.437	0.715	90	82
64	16.0	0.430	0.781	86	80
66	10.0	0.423	0.866	86	78
67	23.5	0.390	0.726	89	82
68	30.0	0.396	0.717	88	81
69	18.5	0.390	0.770	88	81
70	23.0	0.424	0.688	87	80
71	16.5	0.430	0.721	88	81
72	15.0	0.397	0.755	86	79
73	11.5	0.411	0.785	88	80
74	15.0	0.409	0.730	86	80
77	30.0	0.380	0.551	127	118
78	9.5	0.338	0.695	123	116

TABLE XII (Continued)

Run	Pressure Drop - mm Di-butyl Phthalate	Equilibrium Data (Vapor) Inlet y_1	Outlet y_2	Column Temperature Inlet t_1 °C	Outlet t_2 °C
79	10.5	0.373	0.633	162	154
80	10.5	0.404	0.608	160	154
81	10.0	0.386	0.616	163	155
82	10.0	0.395	0.591	162	156
84	5.5	0.386	0.678	162	154
85	5.0	0.403	0.675	161	154
86	5.0	0.405	0.660	161	154
87	10.0	0.435	0.660	184	175
88	10.0	0.435	0.624	187	177
89	7.5	0.440	0.645	187	177
90	4.0	0.450	0.690	184	174
91	9.0	0.518	0.708	208	199
92	14.0	0.516	0.640	209	202
94	2.8	0.483	0.842	206	198
95	11.0	0.467	0.643	211	202
96	5.1	0.458	0.703	208	202
97	5.9	0.447	0.666	208	200
98	7.5	0.448	0.676	209	201
99	4.8	0.448	0.730	208	202
100	11.5	0.427	0.609	212	202
101	8.6	0.370	0.606	210	200
102	6.0	0.362	0.570	213	205

TABLE XII (Continued)

Run	Pressure Drop - mm Di-butyl Phthalate	Equilibrium Data (Vapor) Inlet y_1	Outlet y_2	Column Temperature Inlet t_1 °C	Outlet t_2 °C
103	5.2	0.370	0.635	211	199
104	19.0	0.490	0.670	140	134
105	7.0	0.448	0.797	137	130
106	19.0	0.430	0.647	163	156
107	8.0	0.440	0.698	160	154
108	8.5	0.436	0.775	161	153
109	9.0	0.430	0.715	161	154
110	21.0	0.425	0.630	163	157
111	10.5	0.430	0.685	160	154
112	22.0	0.465	0.715	121	115
113	13.5	0.435	0.733	122	116
114	50.0	0.530	0.730	89	82
115	50.0	0.450	0.484	91	85
116	9.5	0.470	0.580	89	84
117	48.0	0.444	0.544	90	84
118	21.0	0.429	0.585	89	84
119	9.5	0.380	0.568	89	84
120	28.0	0.434	0.720	89	82
121	12.5	0.440	0.632	88	84
122	8.6	0.330	0.400	213	204
123	3.6	0.421	0.577	212	204
124	6.5	0.288	0.434	210	202

TABLE XII (Continued)

Run	Pressure Drop - mm Di-butyl Phthalate	Equilibrium Data (Vapor) Inlet y_1	Outlet y_2	Column Temperature Inlet t_1 °C	Outlet t_2 °C
125	9.5	0.595	0.700	89	84
126	36.0	0.588	0.630	90	84
127	45.0	0.583	0.618	122	117
128	35.0	0.588	0.635	122	117
129	22.0	0.588	0.670	120	114
130	10.0	0.590	0.693	120	114
131	13.0	0.457	0.525	210	203
132	8.0	0.464	0.544	208	201
133	6.0	0.487	0.571	208	200
134	4.0	0.500	0.587	207	199

TABLE XIII

Experimental Results -- Operating Variables

Run	Pressure mm of Hg	Vapor-Liquid Ratio-G/L	Reflux Rate L-lb/secxft^2	Vapor Velocity u_G-ft/sec
1	200	1.00	0.152	2.21
2	200	1.00	0.0936	1.38
3	200	1.00	0.0820	1.21
4	100	1.00	0.0725	2.05
5	100	1.00	0.0585	1.66
6	200	1.00	0.0585	0.863
7	100	1.05	0.100	3.03
8	400	1.00	0.147	1.15
9	400	1.00	0.120	0.930
10	400	1.00	0.0936	0.735
11	400	1.00	0.0704	0.558
12	400	1.05	0.0557	0.460
13	400	1.05	0.0847	0.695
14	400	1.05	0.1225	1.00
17	100	1.05	0.0558	1.67
18	100	1.05	0.0290	0.870
19	100	1.10	0.0639	2.00
20	100	1.10	0.0404	1.26
21	100	1.10	0.0234	0.728
22	50	1.00	0.0702	3.80
24	50	1.00	0.0350	1.91

TABLE XIII (Continued)

Run	Pressure mm of Hg	Vapor-Liquid Ratio-G/L	Reflux Rate L-lb/secxft^2	Vapor Velocity u_G-ft/sec
26	50	1.00	0.0632	3.44
28	50	1.10	0.0320	1.90
29	10	1.00	0.0350	8.80
30	10	1.00	0.0187	4.72
31	10	1.00	0.0117	2.94
32	10	1.00	0.0188	4.69
33	10	1.05	0.0267	7.03
34	10	1.05	0.0222	5.84
35	10	1.05	0.0156	4.09
36	10	1.10	0.0234	6.56
42	50	1.00	0.0515	2.79
43	10	1.00	0.0230	5.75
44	50	1.00	0.0880	4.72
45	50	1.05	0.0669	3.78
46	50	1.05	0.0134	0.778
47	50	1.05	0.0334	1.90
48	50	1.10	0.0383	2.25
49	50	1.10	0.0171	1.03
50	50	1.20	0.0486	3.13
51	50	1.20	0.0312	2.00
52	50	1.20	0.0175	0.940
53	100	1.00	0.0515	1.45
54	50	1.00	0.0258	1.40

TABLE XIII (Continued)

Run	Pressure mm of Hg	Vapor-Liquid Ratio-G/L	Reflux Rate L-lb/secxft^2	Vapor Velocity u_G-ft/sec
55	50	1.10	0.0639	3.76
56	50	1.05	0.0601	3.37
57	50	1.05	0.0289	1.65
58	50	1.00	0.0375	2.02
59	10	1.00	0.0150	3.91
60	10	1.00	0.0210	5.28
61	10	1.05	0.0222	5.84
62	10	1.05	0.0156	4.09
63	10	1.10	0.0213	5.84
64	10	1.10	0.0129	3.50
66	10	1.00	0.0094	2.49
67	10	1.00	0.0210	5.25
68	10	1.00	0.0258	6.40
69	10	1.00	0.0168	4.43
70	10	1.20	0.0159	4.70
71	10	1.20	0.0126	3.78
72	10	1.10	0.0122	3.35
73	10	1.05	0.0104	2.70
74	10	1.20	0.0115	3.42
77	50	1.00	0.0773	4.11
78	50	1.00	0.0281	1.55
79	200	1.00	0.0820	1.22
80	200	1.05	0.0780	1.20

TABLE XIII (Continued)

Run	Pressure mm of Hg	Vapor-Liquid Ratio-G/L	Reflux Rate L-lb/secxft^2	Vapor Velocity u_G-ft/sec
81	200	1.10	0.0704	1.13
82	200	1.20	0.0643	1.14
84	200	1.05	0.0446	0.697
85	200	1.10	0.0404	0.660
86	200	1.20	0.0350	0.621
87	400	1.00	0.129	1.00
88	400	1.20	0.107	0.995
89	400	1.10	0.0937	0.820
90	400	1.10	0.0533	0.460
91	760	1.00	0.168	0.734
92	760	1.00	0.258	1.11
94	760	1.00	0.0608	0.269
95	760	1.00	0.199	0.862
96	760	1.00	0.102	0.491
97	760	1.05	0.111	0.507
98	760	1.00	0.145	0.619
99	760	1.00	0.0983	0.429
100	760	1.05	0.200	0.905
101	760	1.10	0.149	0.702
102	760	1.20	0.102	0.673
103	760	1.10	0.0852	0.404
104	100	1.00	0.0843	2.38
105	100	1.00	0.0351	1.02

TABLE XIII (Continued)

Run	Pressure mm of Hg	Vapor-Liquid Ratio-G/L	Reflux Rate L-lb/secxft^2	Vapor Velocity u_G-ft/sec
106	200	1.00	0.141	2.08
107	200	1.00	0.0655	0.973
108	200	1.00	0.0421	0.634
109	200	1.00	0.0702	1.05
110	200	1.00	0.152	2.30
111	200	1.00	0.0819	1.22
112	50	1.00	0.0608	3.30
113	50	1.00	0.0386	2.10
114	10	1.00	0.0421	9.0
115	10	2.00	0.0211	10.0
116	10	2.00	0.0047	2.28
117	10	1.50	0.0265	9.61
118	10	1.50	0.0125	4.57
119	10	1.50	0.0063	2.28
120	10	1.00	0.0246	6.19
121	10	1.50	0.0078	2.89
122	760	2.00	0.0820	0.680
123	760	1.50	0.0525	0.438
124	760	1.50	0.0858	0.537
125	10	2.00	0.0047	2.33
126	10	2.00	0.0152	7.50
127	50	2.00	0.0525	5.61
128	50	2.00	0.0445	4.80

TABLE XIII (Continued)

Run	Pressure mm of Hg	Vapor-Liquid Ratio-G/L	Reflux Rate L-lb/secxft^2	Vapor Velocity u_G-ft/sec
129	50	2.00	0.0293	3.15
130	50	2.00	0.0152	1.64
131	760	2.00	0.117	0.985
132	760	2.00	0.0760	0.646
133	760	2.00	0.0585	0.448
134	760	2.00	0.0410	0.347

TABLE XIV

Special Correlations and $(HTU)_{OG}$

Run	Product: Reflux Rate and Vapor Velocity; $L \times u_G$	Product: Correlation Factor* and $(L \times u_G)$	Height of Transfer Unit $(HTU)_{OG}$ - ft
1	0.336	0.336	1.82
2	0.129	0.129	1.20
3	0.0992	0.0992	1.15
4	0.149	0.149	1.23
5	0.0972	0.0972	1.12
6	0.0503	0.0503	0.86
7	0.303	0.351	1.55
8	0.169	0.169	1.18
9	0.112	0.112	1.24
10	0.0688	0.069	0.85
11	0.0393	0.039	0.67
12	0.0256	0.029	0.65
13	0.0589	0.066	0.85
14	0.123	0.138	0.97
17	0.0932	0.108	0.85
18	0.0252	0.0292	0.83
19	0.128	0.171	1.09
20	0.0509	0.068	0.93
21	0.0170	0.023	0.71
22	0.267	0.267	1.35

*See Page 54 for definition.

TABLE XIV (Continued)

Run	Product: Reflux Rate and Vapor Velocity; $L \times u_G$	Product: Correlation Factor and $(L \times u_G)$	Height of Transfer Unit $(HTU)_{OG}$ - ft
24	0.0669	0.0669	1.07
26	0.218	0.218	1.25
28	0.0608	0.0857	0.97
29	0.308	0.308	1.41
30	0.0884	0.0884	0.94
31	0.0344	0.0344	0.69
32	0.0881	0.0881	0.84
33	0.188	0.246	1.15
34	0.130	0.170	1.04
35	0.0638	0.0835	0.90
36	0.154	0.260	1.18
42	0.144	0.144	1.15
43	0.132	0.132	1.00
44	0.415	0.415	1.74
45	0.253	0.300	1.63
46	0.0104	0.0124	0.56
47	0.0635	0.0755	1.07
48	0.0862	0.121	1.18
49	0.0176	0.0248	0.73
50	0.152	0.294	1.22
51	0.0624	0.120	1.14
52	0.0164	0.0317	0.72

TABLE XIV (Continued)

Run	Product: Reflux Rate and Vapor Velocity; $L \times u_G$	Product: Correlation Factor and $(L \times u_G)$	Height of Transfer Unit $(HTU)_{OG}$ - ft
53	0.0747	0.0747	1.01
54	0.0362	0.0362	0.67
55	0.240	0.339	1.32
56	0.202	0.241	1.37
57	0.0476	0.0568	0.95
58	0.0758	0.0758	0.85
59	0.0588	0.0588	0.74
60	0.111	0.111	0.99
61	0.130	0.170	1.03
62	0.0639	0.0835	0.80
63	0.124	0.210	1.05
64	0.0451	0.0762	0.74
66	0.0233	0.0233	0.48
67	0.110	0.110	0.96
68	0.165	0.165	1.01
69	0.0745	0.0745	0.81
70	0.0617	0.167	1.01
71	0.0461	0.124	0.86
72	0.0409	0.069	0.76
73	0.0310	0.0406	0.63
74	0.0393	0.106	0.76
77	0.318	0.381	1.80

TABLE XIV (Continued)

Run	Product: Reflux Rate and Vapor Velocity; $L \times u_G$	Product: Correlation Factor and $(L \times u_G)$	Height of Transfer Unit $(HTU)_{OG}$ - ft
78	0.0436	0.0436	0.81
79	0.100	0.100	1.00
80	0.0935	0.107	1.23
81	0.0795	0.102	1.04
82	0.0733	0.118	1.14
84	0.0310	0.0355	0.81
85	0.0266	0.0341	0.84
86	0.0217	0.0350	0.79
87	0.129	0.129	1.02
88	0.106	0.161	1.03
89	0.0768	0.095	1.03
90	0.0245	0.0304	0.82
91	0.123	0.123	1.00
92	0.286	0.286	1.67
94	0.0164	0.0164	0.45
95	0.172	0.172	1.21
96	0.501	0.501	0.77
97	0.0564	0.0620	0.91
98	0.0886	0.0886	0.93
99	0.0420	0.0420	0.69
100	0.181	0.199	1.10
101	0.105	0.127	0.83

TABLE XIV (Continued)

Run	Product: Reflux Rate and Vapor Velocity; $L \times u_G$	Product: Correlation Factor and $(L \times u_G)$	Height of Transfer Unit $(HTU)_{OG}$ - ft
102	0.0667	0.0960	0.88
103	0.0345	0.0417	0.70
104	0.200	0.200	1.37
105	0.0358	0.358	0.65
106	0.294	0.294	1.17
107	0.0638	0.0638	0.93
108	0.0267	0.0267	0.63
109	0.073	0.073	0.84
110	0.350	0.350	1.37
111	0.0996	0.100	0.96
112	0.200	0.200	1.10
113	0.081	0.081	0.91
114	0.380	0.380	1.50
115	0.211	9.5	9.2
116	0.0107	4.82	1.58
117	0.255	2.37	3.16
118	0.057	0.53	1.53
119	0.0144	0.134	1.03
120	0.152	0.152	1.13
121	0.0225	0.208	1.12
122	0.0558	0.223	1.25
123	0.0230	0.0517	0.70

TABLE XIV (Continued)

Run	Product: Reflux Rate and Vapor Velocity; $L \times u_G$	Product: Correlation Factor and $(L \times u_G)$	Height of Transfer Unit $(HTU)_{OG}$ - ft
124	0.0461	0.104	1.10
125	0.0109	0.470	1.97
126	0.114	5.10	5.9
127	0.366	4.43	6.8
128	0.214	2.58	4.6
129	0.0923	1.12	2.1
130	0.0249	0.289	1.29
131	0.115	0.460	1.88
132	0.0490	0.196	1.21
133	0.0262	0.105	1.00
134	0.0142	0.056	0.78

TABLE XV

Experimental Results - Total Reflux Runs: Product of Vapor Velocity and Square Root of Pressure (Measured in Atmospheres)

Run	u_G	Run	u_G
1	1.13	68	0.736
2	0.708	69	0.510
3	0.620	77	1.06
4	0.745	78	0.398
5	0.604	79	0.626
6	0.441	87	0.725
8	0.834	91	0.734
9	0.675	92	1.11
10	0.534	94	0.269
11	0.405	95	0.862
29	1.01	96	0.491
30	0.542	98	0.619
31	0.337	99	0.429
32	0.539	104	0.866
42	0.744	105	0.372
43	0.661	106	1.08
44	1.21	107	0.500
53	0.527	108	0.325
54	0.360	109	0.539
58	0.519	110	1.18
59	0.450	111	0.626
60	0.606	112	0.847
66	0.286	113	0.540
67	0.604	114	1.03

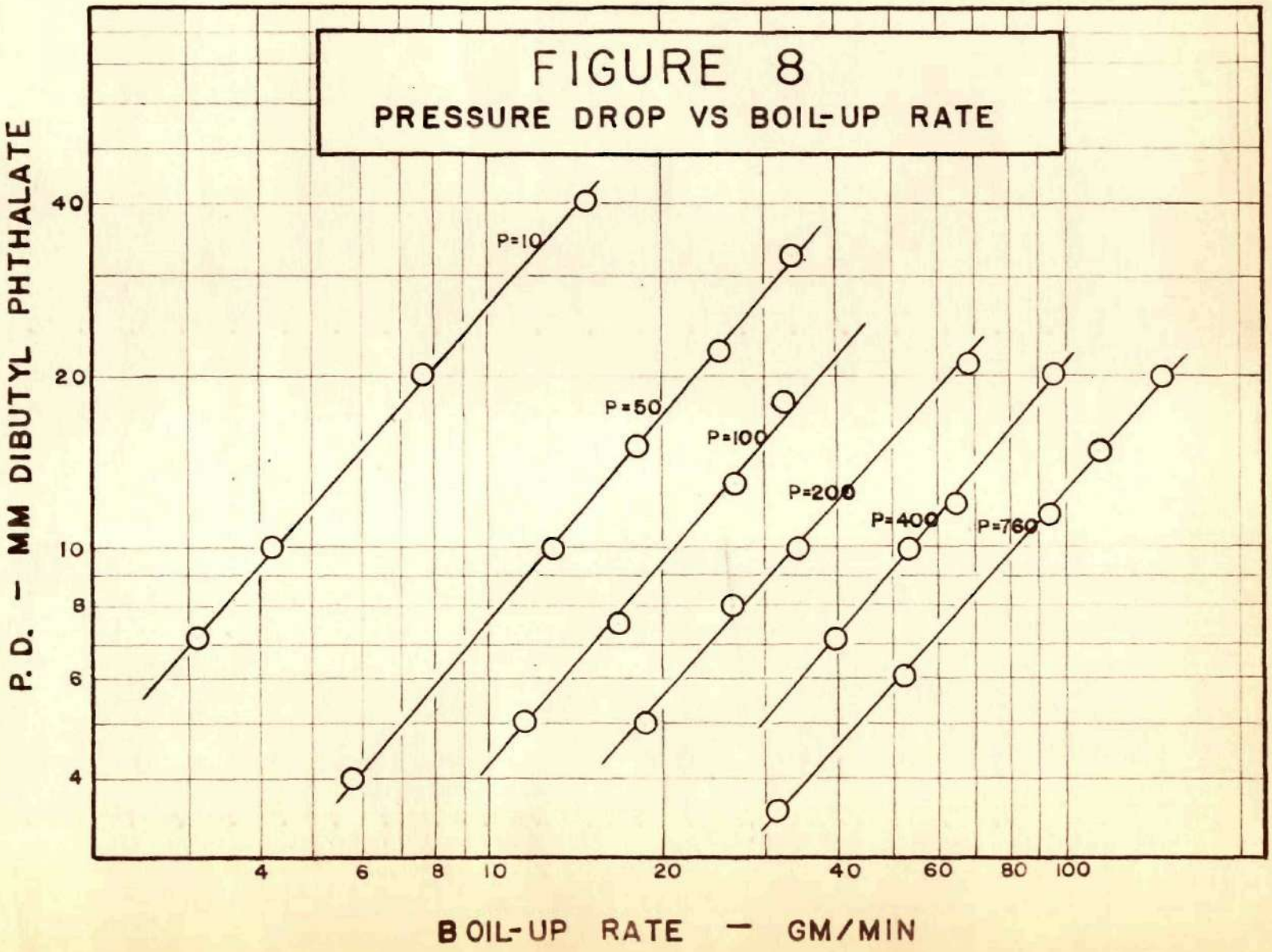
FIGURE 8
PRESSURE DROP VS BOIL-UP RATE
P. D. - MM DIBUTYL PHTHALATE
40
20
10
8
6
4
P=10
P=50
P=100
P=200
P=400
P=760
4
6
8
10
20
40
60
80
100
BOIL-UP RATE - GM/MIN

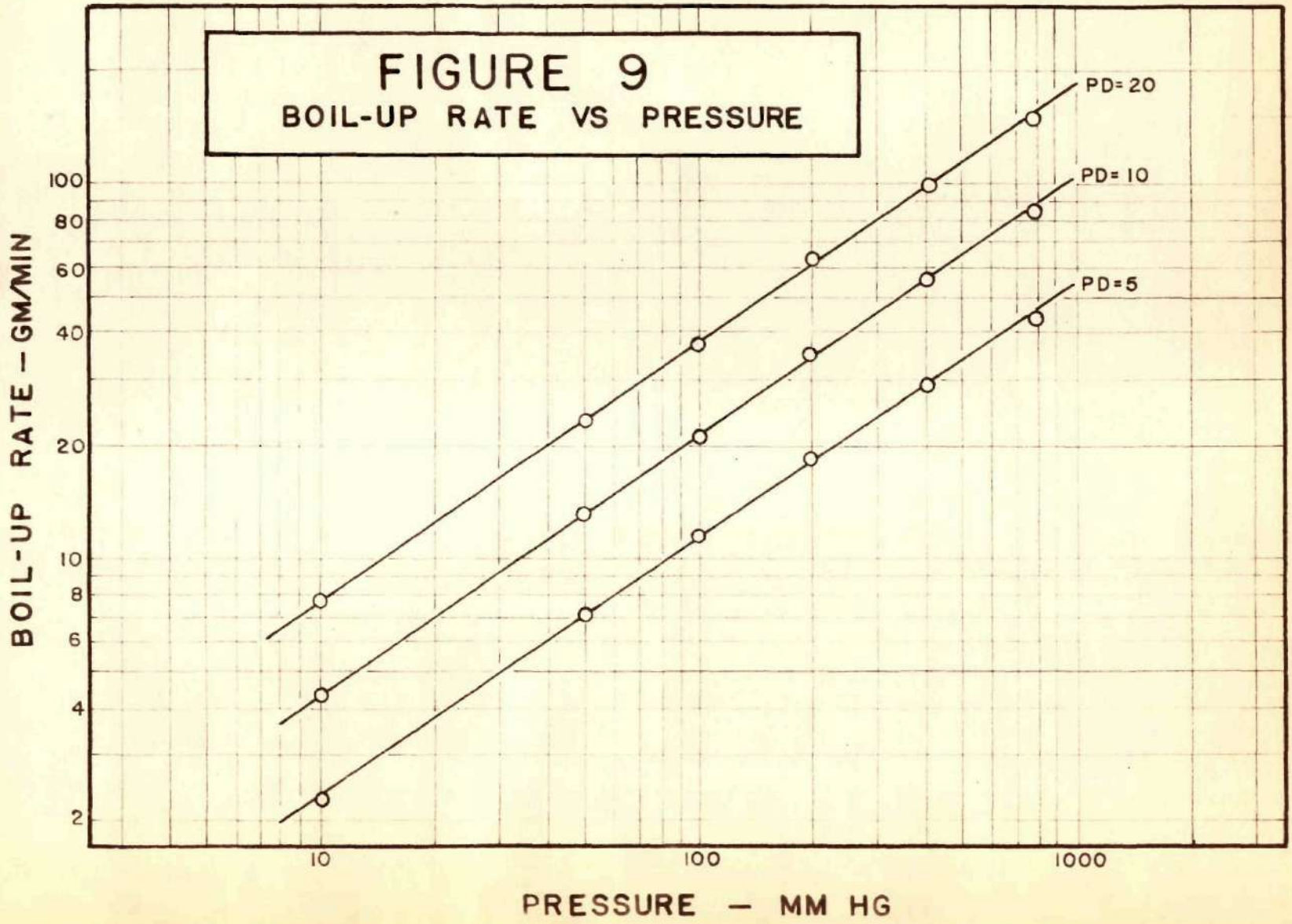
FIGURE 9
BOIL-UP RATE VS PRESSURE
PD=20
PD=10
PD=5
BOIL-UP RATE – GM/MIN
100
80
60
40
20
10
8
6
4
2
10
100
1000
PRESSURE – MM HG

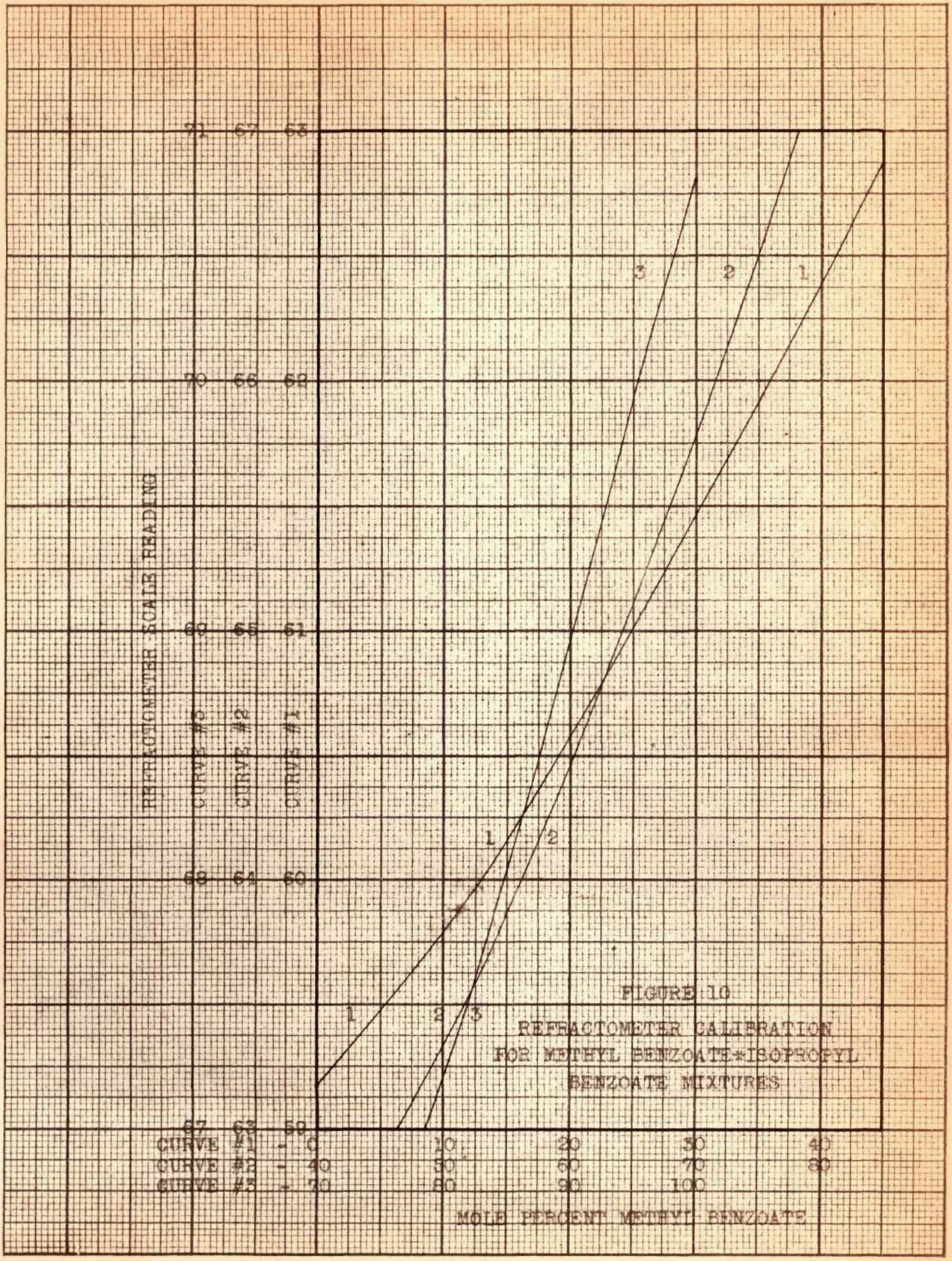

FIGURE 10

REFRACTOMETER CALIBRATION FOR METHYL BENZOATE*ISOPROPYL BENZOATE MIXTURES